FRP PRODUCTS USING RUBBER MOLDING TECHNIQUE

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By

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CERTIFICATE

It is certified that the work contained in the thesis entitled "**FRP Products Using Rubber Molding Technique**", *by* Suraj Kumar Behera, *has been carried out under our supervision, and this work has not submitted else where for a degree.*

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SURAJ KUMAR BEHERA

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Dedicated to

MY PARENTS

ABSTRACT

A rubber molding technique is used to prepare a fiber reinforced polymer product from glass fiber and epoxy resin. The technique is based on matching die set, where die is made from hard metal like steel and punch from flexible material like rubber. The use of flexible rubber punch applies hydrostatic pressure on the surface of the product. A split steel die and rubber punch is designed and fabricated to prepare FRP product (pump cap). The same split steel die is also used to cast rubber punch. Four varieties of rubber materials are used to prepare rubber punch. These are (i) natural, (ii) butyl (iii) silicon and (iv) polybutadiene rubbers. Polyester resin does not cure with any of the above rubber, but epoxy resin cures well with all of the above rubber.

Quality tests like burn test, coin test and microstructure study have been done on the products to find out void content, presence of delamination and bonding between fiber and resin. It is found that product made by rubber molding technique using above four varieties of rubber punch has void content less than 3 %, free of delamination and better bonding between fiber and resin.

Mechanical tests like interlaminar fracture toughness test, interlaminar shear test and tension test have been conducted on appropriately designed specimen to characterize the product made by conventional method and rubber molding technique. It is found that the specimens prepared by rubber molding technique using butyl and silicon rubber sheet have interlaminar fracture toughness equivalent to those of specimen prepared by the conventional method. But the specimen prepared by using natural rubber and polybutadiene found to have significantly lower interlaminar fracture toughness by 21% and 17% respectively compared to the specimen prepared by conventional method. In interlaminar shear test, specimen prepared using natural and polybutadiene rubber sheets has significantly lower interlaminar shear strength by 57% and 24% respectively

than the specimens prepared by conventional method. However specimens prepared by using butyl and silicon rubber sheets found to have marginally higher values of interlaminar shear strength compared with specimens prepared by conventional method. In tension test, specimens made by rubber molding method have marginally better value of tensile strength than specimen prepared by conventional method except specimens prepared by natural rubber, whose tensile strength is equivalent to that of specimens prepared by conventional method. The elastic modulus of the specimen prepared by rubber molding technique using butyl and silicon rubber sheets found to have marginally higher value by 9 % each than the specimens prepared by conventional method. Thus, the most recent method "rubber molding technique" to fabricate FRP gives comparable mechanical properties than that of conventional method. But the FRP product made by using natural rubber gives inferior mechanical properties than that of polybutadiene, butyl and silicon rubber.

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Fiber reinforced polymer (FRP) is an important class of engineering materials and gaining popularity amongst the designer due to its unique properties like high specific tensile strength, high specific modulus, corrosion resistance, high fatigue strength, etc. FRP consists of a discontinuous fiber phase reinforced in a continuous phase of polymer. The fibers used are glass, graphite, Kevlar, etc and the polymer can be thermoplastic or thermosetting. Thermosetting polymers are used, where strength and high operating temperature are important considerations. Thermosetting polymers used for preparing FRP are polyester resin, epoxy resin, phenolic, etc. These FRP composites are being used in applications like aerospace, automobile, sports goods, railways, fuel cylinders, support beams of highways, bridges, paper making rollers, wind turbine blades, recreation industries, etc.

1.2 MANUFACTURING OF FRP PRODUCTS

Manufacturing of FRP is mainly based on casting. The casting of FRP product is more difficult than casting of metal, because liquid metal has good flowability and can easily flow in gating channel to fill the cavity. During manufacturing of FRP components, polymer being liquid with low viscosity has good flowability, where as fibers have high stiffness and do not take shape easily over high curvature of FRP components. Several methods have been developed to manufacture the FRP products [Kumar, 2000: Parkyn, 1970: Agarwal and Broutman, 1990; Hollaway, 1994; Kumar et al, 1995; Mangalgiri, 1999, Sah, 2002]. Some of these are filament winding, pultrusion method, vacuum bagging technique, autoclave technique, matching die set compression molding, resin transfer molding, rubber pressure molding, etc.

Mallon and Obradaigh [1988] have developed a pilot experimental autoclave for polymeric diaphragm forming of continuous fiber reinforced thermoplastic composite. An autoclave is heated using standard heater and is pressurized by compressed nitrogen gas. Thin section tolling is successfully developed to investigate forming over a 90° single curvature bend.

Stringer [1989] has investigated wet lay up/ vacuum bag technique and optimized the fabrication of carbon fiber epoxy composites with high fiber volume fraction and low void content by incorporating a dwell period at the start of cure cycle to increase the resin viscosity before applying the bagging pressure.

Application of hydrostatic pressure during preparation of FRP gives better and uniform properties though out the component. The manufacturing process, which gives hydrostatic pressure during curing of perform are vacuum bagging technique, autoclave technique, resin transfer molding and rubber pressure molding. For providing hydrostatic pressure in vacuum bagging technique a vacuum pump is used to compress the prepreg by one atmospheric pressure through out the FRP component. Autoclave technique is similar to vacuum bagging technology with an addition of atmosphere of compressed nitrogen inside the autoclave. Recently Sah [2002] has developed rubber pressure molding technique, which uses a matching die set to prepare FRP component, where the die is made of hard metal like steel and the punch is made of a flexible material like rubber. Rubber pressure molding has several advantages over conventional methods of matching die set, where punch and die are made of hard metal like steel.

1.3 CHARACTERIZATION OF FRP PRODUCTS

The failure of FRP composites may be observed in many forms like (i) micro cracking of matrix (ii) breaking of fibers (iii) delamination (iv) debonding etc. These failures may occur separately or jointly [Agarwal and Broutman, 1990].

Delamination may be in the form of separation of plies under mechanical, thermal or environmental loading. Geometric discontinuities and manufacturing flaws are the factors for initiating an interlaminar crack. Such problems are not observed in metals. Impact on the laminates also leads to delamination. Keary et al [1985] have used double cantilever beam [DCB] specimen to measure interlaminar fracture toughness in mode I. They have analyzed the data through various schemes and compared with one another.

To characterize the behavior of delamination and fracture analysis for separation modes, Wilkins et al [1982] have found a critical energy release rate for Mode I and Mode II delaminations. They have used DCB specimen for Mode I and cracked lap shear (CLS) specimen for Mode II.

Devitt et al [1980] have developed a nonlinear theory for energy release rate using double cantilever beam. But the interlaminar fracture toughness of glass fiber reinforced in polyester matrix composite using width tapered double cantilever beam specimen have been found by Han and Koutsky [1981]. Mall et al [1987] have used DCB specimen to investigate the effect of loading rate on interlaminar fracture toughness.

Lantz [1969] has used sandwich specimen for determining off axis and transverse properties. Pagano and Halpin [1968] suggested that a uniform state of stress and strain exist at the center of an off-axis tensile specimen if length to width ratio is sufficiently large.

Waddoups et al, [1968] and Hill [1968] have found tensile properties of composite using a sandwich beam subjected to bending, where a thin layer of composite material is bonded on the top and bottom of a thick substrate such as aluminum honey-comb. One side of the composite is loaded in tension and other side in compression. Thus, a single specimen can be used to determine both tensile and compressive properties simultaneously. Agarwal and Broutman [1990] have described the details on uniaxial tension test.

Interlaminar shear strength as an important property for composite laminates subjected to bending. This has been recognized by Pagano and pipes [1973]. They have observed experimentally the phenomenon of high interlaminar normal and shear stress occurring in a region near to the free edge of composite laminates subjected to in-plane loading. Rybicki [1971] has computed the interlaminar stresses using finite element difference approach. But the finite element technique as well as finite difference does not give a good estimate of stress in this region due to the high stress gradient near to the free edge. But Hsu and Herakovich [1976.1977] have analyzed the problem of finite element analysis by dividing each layer into an inner and outer region. The inner region is analyzed by the classical laminates theory. The region near the free edge is analyzed by using a perturbation technique and concluded that their results provide better estimate than finite difference solution given by Pipes and Pagano [1970].

1.4 OBJECTIVE OF THE THESIS

All existing techniques for fabrication of FRP products have some disadvantages. In present work, a recently developed technique known as "Rubber Molding Technique" is used to fabricate FRP products. This technique uses a metal die and a flexible rubber punch to fabricate FRP products, which enables the application of nearly hydrostatic pressure on the component during curing of a preform. A FRP component "Pump Cap" is prepared and various quality tests have been conducted to check the suitability of rubber molding technique using four varieties of rubber punches (natural, butyl, and silicon and polybutadiene rubber). A comparison of different mechanical properties of specimens prepared by conventional matching die set and rubber pressure molding (using above four varieties of rubber sheets) has been done.

1.5 THESIS LAYOUT

The first chapter (Chapter 1) deals with the introduction to FRP composites and various existing manufacturing process and testing of FRP composites are presented.

The methods of fabrication of FRP which enables application of hydrostatic pressure on the component during curing and advantages of recently developed technique (rubber pressure molding) are described in chapter 2.

Chapter 3 illustrates the various curing tests of rubber to check the feasibility of rubber in a resin system.

In Chapter 4, fabrication details are discussed to prepare the product, "Pump Cap". Die design and casting process of rubber punches are described. Also, process to make FRP composite (pump cap), quality testing and comparison of products are described.

Interlaminar fracture toughness tests are presented in Chapter 5. In Chapter 6, details of interlaminar shear tests are given. Chapter 7 discusses the tensile test.

In Chapter 8, major conclusions of present work are discussed.

CHAPTER 2
RUBBER MOLDING TECHNIQUE

2.1 INTRODUCTION

Manufacturing of FRP products is mainly based on casting. Casting of FRP components is more difficult than casting of metal, because liquid metals has good flowability and it can easily flow in gating channel to fill cavity. During manufacturing of FRP components, polymer being liquid with low viscosity has good flowability but fibers have high stiffness and do not drape easily over high curvature of FRP components. Therefore application of pressure is an important parameter to provide shaping of material before solidification of polymer. When the developed pressure applied is hydrostatic, it serves several functions as follows:

(i) It helps to maintain product uniformity within the part.

(ii) It removes entrapped air bubbles.

(iii) It improves wetting between fiber and resin.

(iv) It prevents shifting of fiber orientation during cure.

Existing techniques that provides hydrostatic pressure during curing of FRP

A flexible medium is essential for utilizing hydrostatic pressure during curing of FRP product. Few existing and widely used techniques like bagging technique, autoclave technique, resin transfer molding, etc use hydrostatic pressure.

(1) Bagging technique

In this technique, prepregs are cut to the required shape/dimension and stacked together to make a preform. Then perform is bagged and vacuum is created inside the bag, which results into compression of prepregs by an atmospheric pressure. Fig 2.1 shows the

schematic diagram of this process. After applying the release agent inside the die, preform of the product, perforated separator, bleeder and vacuum bag are stacked. When vacuum inside the bag is created, the bag gets compressed and the vacuum is maintained till the product is cured. Maximum pressure generated in the preform is one atmospheric pressure, which is some time inadequate. This is a major limitation of this process. But this technique is relatively simple and inexpensive, because an autoclave is not required. However, the bag is thrown away after fabrication of each piece.

(2) Autoclave technique

This is similar to the bagging technique till creation of the bag i.e. stacking of various materials over the die. The difference is that entire assembly is kept in an autoclave. In the autoclave an atmosphere of compressed nitrogen gas is generated, thus, high pressure can be applied on the preform. Furthermore, vacuum is created inside the bag to bleed out air and volatile gases inside the bag. The schematic diagram of this technique is shown in Fig 2.2. Although this technique works efficiently but it has some limitations. Expensive tolling is required and after using the bag once entire bagging material is disposed off. This technique is required in making airplane components where high production rate is not required but quality of the component is a primary concern.

(3) Resin transfer molding

In this technique, dry (i.e., unimpregnated) reinforcement is pre-shaped and oriented into a skeleton of actual part known as the preform, which is inserted into matched die mold. The mold is then closed, and a low viscosity resin is injected into the tool. The air is displaced and escapes from vent port placed at high points. During this time, it is known as the injection or infiltration stage, in which resin wets out the fiber. Heat applied to the mold activates polymerization mechanisms that solidify the resin in the step known as cure. The resin cures during filling and continued after the filling process. Once the part develops sufficient green strength, it is demolded. This process is schematically shown in Fig 2.3.

2.2 RUBBER MOLDING TECHNIQUE

This technique is recently developed by Sah, 2002. This process is similar to matching die set method. But lower die is made from hard metal and upper die (punch) is made from a flexible rubber material. Because the punch is flexible, it enables the application of nearly hydrostatic pressure on the component during curing of FRP component. The schematic diagram of this technique is shown in Fig 2.4. A releasing agent on the die is applied to prevent adhesion of FRP component to the metal die. Resin wetted fiber reinforcement (fabric, mat or unwoven fibers) is cut and laid up in the tool. Each layer has a pre-specified direction to optimize stiffness and other characteristics. After staking all the layers, the flexible rubber punch is placed over the preforms. The closed tool is kept in press, which applies pressure to the composite at room temperature. Die and punch are designed to remove air bubbles and bleed out excess resin from the composite during application of pressure.

Rubber and resin both being polymers, There is possibility of some chemical reaction between these two materials at operating temperature and pressure. Therefore before selection of rubber punch and resin system curing properties of resin system on the surface of rubber is essential to check. This can be done by placing resin over surface of the rubber and left it for specified time to check its curing properties.

Single hard mold can be used to prepare rubber punch and FRP component of thin wall product as rubber is flexible enough to small deformation. This method can be used to fabricate fiber reinforced plastic product for aerospace and automobile industries.

2.3 ADVANTAGES OF RUBBER MOLDING TECHNIQUE

The advantages of this technique are as follows:

(i) Developed pressure applied is hydrostatic even on vertical walls, which enables to maintain product uniformity within single part.

(ii) Use of single hard mold.

(iii) Bagging materials are not used.

(iv) Tooling cost is low.

(v) Rubber punch can be made from the same mold using an appropriate spacing material (for thin wall products of wall thickness less than 1.5 mm, spacing material is not required as rubber is flexible enough to deform).

(vi) Same rubber punch can be used several times.

2.4 CLOSURE

The rubber molding technique is one more alternative to the pressure bag. Due to the application of nearly hydrostatic pressure, product uniformity is able to be maintained. Fabrication of rubber punch is easy and prepared at a temperature of 150°C and pressure of 8 MPa. Various advantages of this technique reduces the manufacturing cost of FRP for industries like aerospace, automotive, marine, recreation, electrical, construction, agriculture, etc.

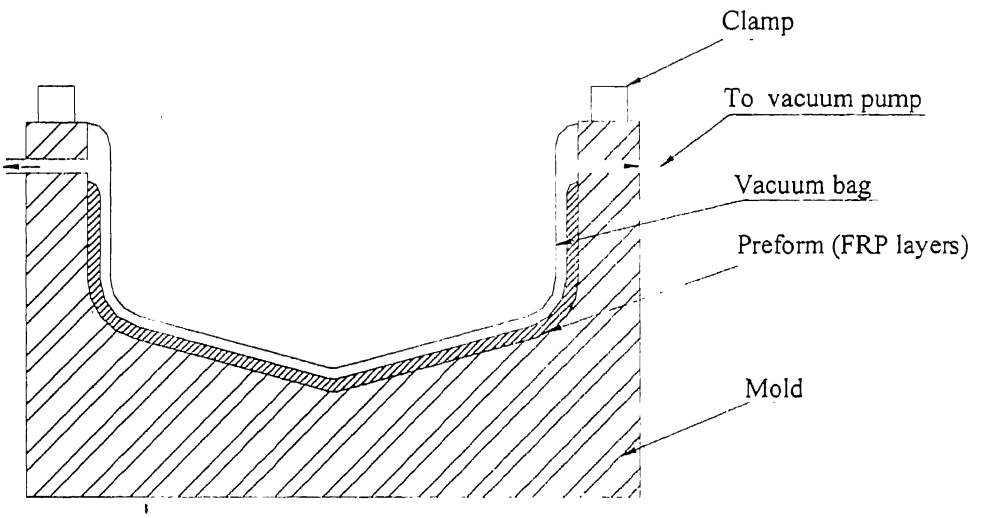


Fig. 2.1: Vacuum-bag molding

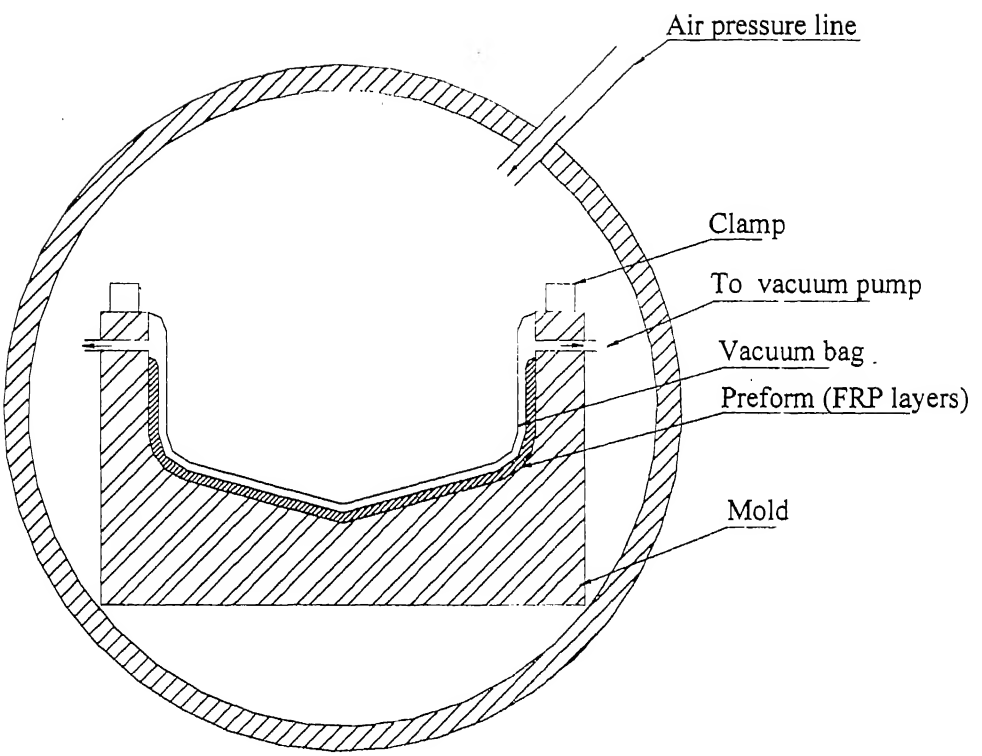


Fig. 2.2: Autoclave technique

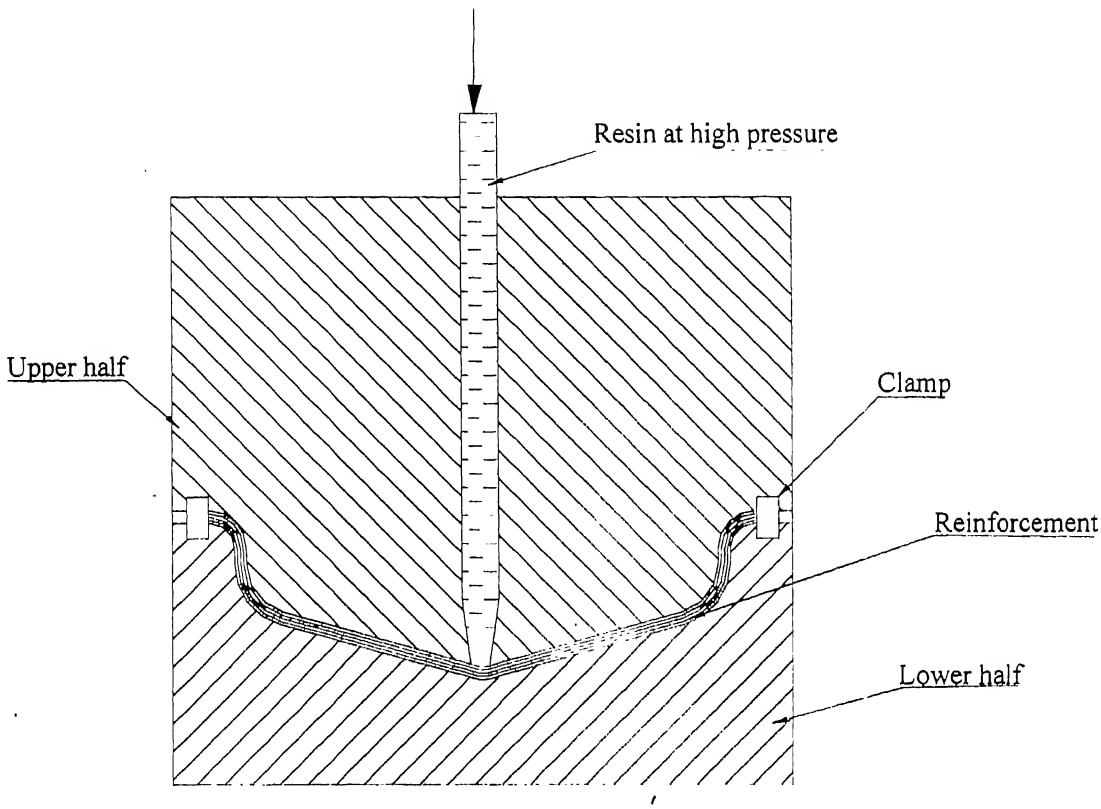


Fig 2.3: Resin transfer molding

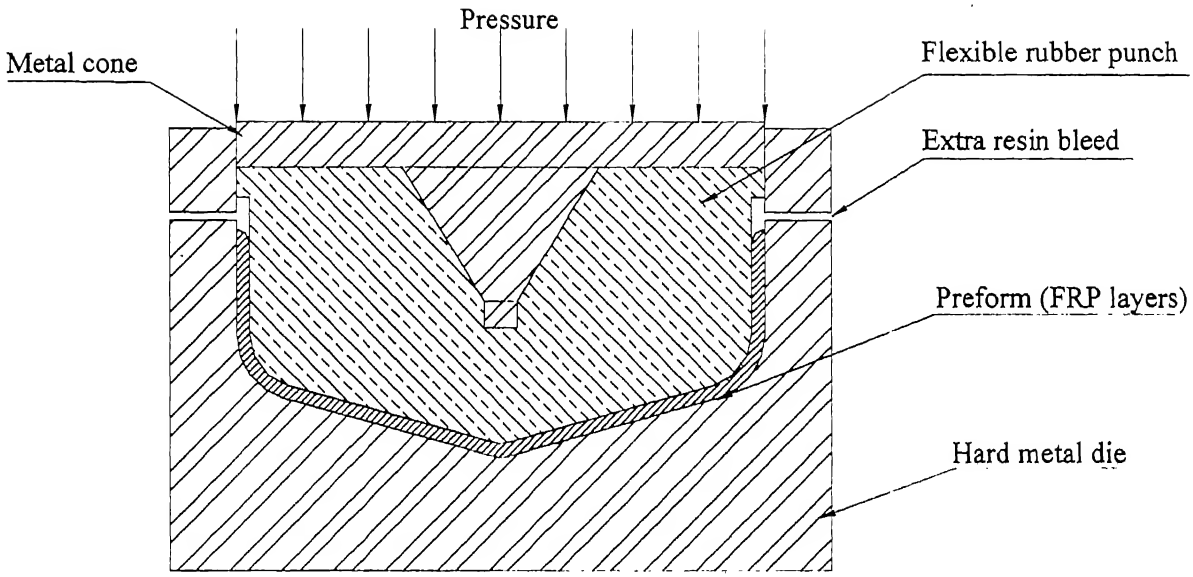


Fig 2.4: Rubber molding technique

CHAPTER 3

CURING TEST OF POLYESTER AND EPOXY RESIN WITH RUBBER

3.1 INTRODUCTION

Before making FRP it is essential to check the curing properties of resin with the surface of mould and punch for better surface finish as well better quality. Sah [2002] has found that polyester resin is not curing with the natural rubber. To find out the reason, numbers of experiments have been conducted on polyester and epoxy resin using four verities of rubbers. These are (i) natural rubber, (ii) butyl rubber (iii) silicon rubber, and (iv) polybutadiene rubber. In the present work curing properties of polyester and epoxy resin on the surface of rubber are tested. Polyester resin and epoxy resin is used on rubber surface without applying any coating agent to check its curing properties. Also polyester resin is used on rubber with a coating agent over its surface. Cured rubber is a mixture of several chemicals. To find out the chemical present in cured rubber that decreases curing rate of polyester resin, different chemicals are tested separately with polyester resin.

3.2 CURING TESTS WITH RAW RUBBER

In this experiment polyester resin is tested on the surface of raw rubbers. Four varieties of rubbers (i) natural, (ii) butyl, (iii) silicon and (iv) polybutadiene rubbers are used to check curing property of polyester resin and epoxy resin. Rubbers are cut in the shape of a disc with dimensions Φ53 mm X 10 mm thickness and kept in the set up shown in Fig 3.1. The set up consists of a steel cylindrical tube with internal diameter of 53 mm, thickness of 4 mm and length of 10 mm welded on a square plate with dimension 100 X100 X 6 mm. Polyester and epoxy resin is tested by pouring over the rubber surface. Polyester resin poured over rubber is mixed with catalyst (benzole peroxide) and hardener (methyl ethyl ketone). The weight ratio of polyester resin, catalyst and hardener is 100:2:2.

Similarly epoxy resin is mixed with hardener (HY951) with a weight ratio of 100:10. Then set up is left for 24 hours to check their curing behaviors on the surface of rubber. The results of this experiment are given in Table 3.1.

Table 3.1: Detail of experimental results of polyester and epoxy resin on the surface of raw rubber

S.No	Rubber material	Resin used	Curing result on the surface of rubber
1	Raw natural rubber	Polyester	Not cured
2	Raw butyl rubber	Polyester	Not cured
3	Raw silicon rubber	Polyester	Not cured
4	Raw polybutadine	Polyester	Not cured
5	Raw natural rubber	Epoxy	Cured well
6	Raw butyl rubber	Epoxy	Cured well
7	Raw silicon rubber	Epoxy	Cured well
8	Raw polybutadine	Epoxy	Cured well

3.3 CURING TESTS WITH POLYESTER RESIN ON CURED RUBBER

This section describes various tests to find out curing properties of polyester resin on the surface of cured rubber. Tests are conducted with and without applying a release agent on the rubber surface.

Observing difficulties of curing with polyester resin on the raw rubber surfaces, cured rubber is prepared by mixing chemicals i.e. zinc oxide, stearic acid, accelerator and sulphur to test their curing properties with polyester resin. The mixing of chemicals on raw rubber is done on "two roll mixing mill". After mixing all the chemicals with rubber, the uncured rubber is placed in the setup shown in Fig 3.2. The set up consists of a steel cylindrical tube placed between two steel square plates. The set up is kept in a hydraulic press at a temp of 150°C and pressure of 2 MPa for 20

minutes and allowed to cure to form a solid rubber block (Φ 53 mm X 10 mm). Then cured rubber block is placed on the bottom of the setup shown in Fig 3.1. Polyester resin along with catalyst and hardener is poured over it and left for 24 hours to check its curing properties.

To check curing properties of polyester resin with a coating agent on cured rubber surface, a thin layer of water based mold releasing agent "Safelease 30" (Supplied by Airtech International Inc., California USA) is applied on the surface of rubber block. The safelease 30 is TEFLON. After applying a thin coating of release agent on the rubber block, it kepts at a room temperature to evaporate water and deposit a thin layer of teflon as a residue on the surface of rubber block. Then rubber block with coating of release agent is placed on the bottom of the setup shown in Fig 3.1. Polyester resin along with catalyst and hardener is poured over it and left for 24 hours to check its curing properties on the surface of rubber. The final result of this experiment is given in Table 3.2.

Table 3.2: Detail of experimental results of polyester resin over cured rubber surface

S.No	Rubber material	Curing result on the surface of rubber
1	Cured natural rubber	Not cured
2	Cured butyl rubber	Not cured
3	Cured silicon rubber	Not cured
4	Cured polybutadine	Not cured
5	Cured natural rubber (with coating agent on surface)	Cured well
6	Cured butyl rubber (with coating agent on surface)	Cured well
7	Cured silicon rubber (with coating agent on surface)	Cured well
8	Cured polybutadine (with coating agent surface)	Cured well

3.4 CURING TESTS WITH EPOXY RESIN ON CURED RUBBER

This section describes the test to find out curing properties of epoxy resin on the surface of cured rubber. A cured rubber block (Φ 53 mm X 1.5 mm) is placed on the bottom of the setup shown in Fig 3.1. Epoxy resin along with hardener is poured over it and left for 24 hours to check its curing properties on the surface of rubber. The final results of this experiment are given in Table 3.3.

Table 3.3: Detail of experimental results of epoxy resin over cured rubber surface without coating agent

S.No	Rubber material	Curing result on the surface of rubber
1	Cured natural rubber	Cured well
2	Cured butyl rubber	Cured well
3	Cured silicon rubber	Cured well
4	Cured polybutadine	Cured well

3.5 CURING TESTS WITH POLYESTER RESIN ON DIFFERENT CHEMICALS USED IN RUBBER

Cured rubber is a mixture of several chemicals. These are steric acid, zinc oxide, clay, silica, carbon black, sulphur, accelerators, etc. The purpose of using these chemicals in the rubber is to enhance mechanical properties. But some of these chemicals present in the cured rubber decrease the curing rate of polyester resin. To identify, which chemical in rubber effects curing of polyester resin, a new set-up shown in Fig 3.3 is developed here. The set up consist of a central blind hole of Φ 28 mm on a steel block of dimension 55 X 35 X 30 mm. On the bottom side of block one M3 hole is made to remove the solid chemical and resin block easily with the help of a bolt. Each chemical out of steric acid, zinc oxide, clay, silica, carbon black, sulphur, curing agents and accelerators is heated to

slightly above its melting point in setup (shown in Fig 3.3) and then allowed to cool down to become solid block. On the top of the solid block polyester resin along with catalyst and hardener is poured and left for 24 hours to check their curing behavior on the surface of solid chemical block. This procedure is repeated for all the chemicals used to make rubber. The results of this experiment are given in Table 3.4

Table 3.4: Detail of experimental results of polyester resin over chemical ingredients

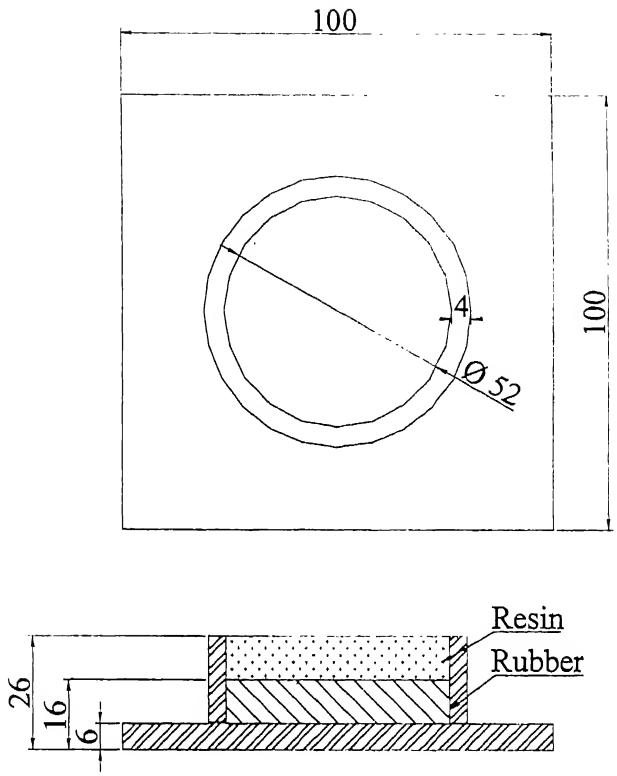
S.No	Chemicals used	Curing result on the surface of chemical block
1	Zinc oxide	Cured well
2	Steric acid	Cured well
3	Clay	Cured well
4	Carbon black	Cured well
5	Sulphur	Cured well
6	Accelerator	Cured well

3.6 RESULTS AND DICUSSION

Several experiments have been conducted on polyester and epoxy resin using four verities of rubbers i.e. (i) natural rubber, (ii) butyl rubber (iii) silicon rubber, and (iv) polybutadiene rubber. Also some chemicals like steric acid, zinc oxide, clay, silica, carbon black, sulphur, accelerators used to enhance mechanical properties of rubber are tested with polyester resin to identify, which chemical in the rubber effects curing of resin. It is clear from Tables 3.1 to 3.4 that the polyester resin is not curing on any one of the raw rubber surfaces used in this experiment. However polyester resin cures well on the surface of rubber with a coating of release agent. Epoxy resin cures well on the surface of all rubbers used in this experiment. The chemicals used in rubber, listed in Table 3.4 are not causing any problem in curing of polyester resin on the surface of rubber.

3.6 CLOSURE

Polyester resin is not curing with any rubbers used in the present work, i.e. natural, butyl, silicon and poly butadiene rubbers, but epoxy resin cures perfectly. So epoxy resin is used as a matrix material for the present work.



3.1: Set-up for testing curing properties of rubber

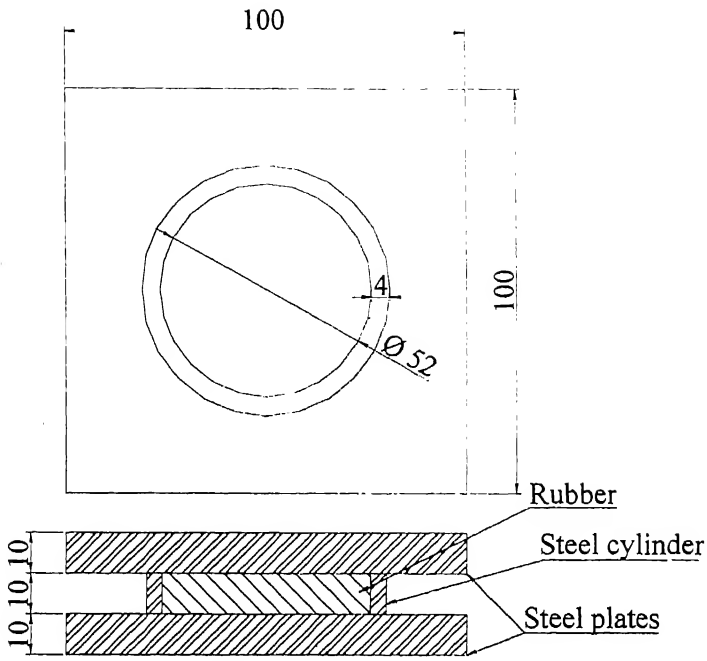


Fig 3.2: Set-up for preparation of rubber block under hydraulic press

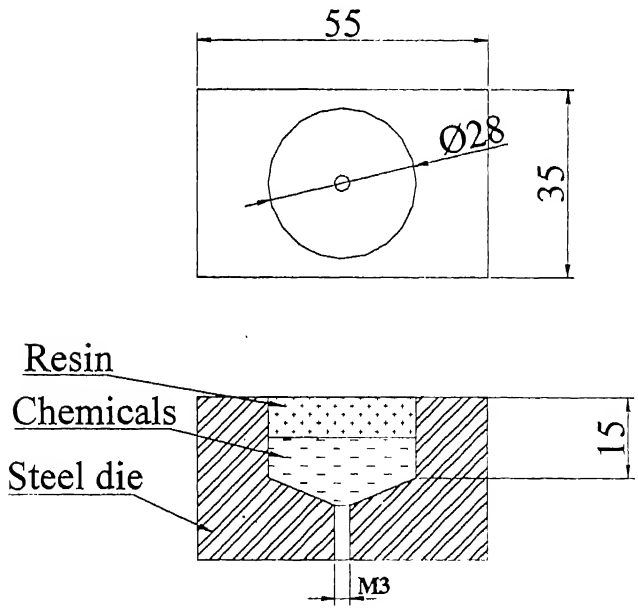


Fig 3.3: Set-up for curing test of rubber ingredients

CHAPTER 4
FABRICATION: COMPONENT AND TEST SPECIMEN

4.1 INTRODUCTION

A component, pump cap (Fig 4.1) has been chosen to be made from FRP using rubber molding technique. Tooling required for molding the component is discussed in this chapter.

The component is made by using four varieties of rubber punches. These are (i) natural rubber (ii) butyl rubber (iii) silicon rubber and (iv) polybutadiene rubber. To understand the effectiveness of each rubber material on quality of the resulting component, specimens are prepared. The details of preparing the specimen are is described in this chapter. Various test like interlaminar fracture toughness test, interlaminar shear test and tension test are discussed in the subsequent chapters.

4.2 PRODUCT DEFINITION

The pump cap selected in this study, which is a component of cooler pump is usually made of steel sheet of thickness 1 mm. It usually gets rusted and it was felt that a component of composite pump may be a more appropriate material. This component has three important geometry elements (i) cylindrical, (ii) conical and (iii) flat surface (Fig 4.1). The cylindrical part has an outer diameter of 120 mm and thickness of 1.5 mm; the conical portion has a half cone angle of 45^{o} and thickness of 1.5 mm; the flat portion has a diameter of 70 mm and thickness of 1.5 mm. The total height of the pump cap is 75 mm. The glass fiber fabric/epoxy resin is used to fabricate this component.

4.3 CONFIGURATION OF RUBBER MOLDING TECHNIQUE

To prepare pump cap from GFRP (Glass Fiber Reinforced Polymer) a split steel die, steel cone and flexible rubber punch are used. A schematic diagram of the overall setup is shown in Fig 4.2. Split steel die has a interior conforming to a external shape of the component. The die is made in two parts for easy removal of component once it is cured. A perform of glass fiber fabrics wetted with epoxy resin is placed in between split die and rubber punch as shown in Fig 4.2. Then the rubber punch is pressed by a suitably designed steel cone. A force on the steel cone is applied by the hydraulic press. The product is cured in 16 hours at room temperature and pressure of 0.4 MPa.

4.4 PREPARATION OF STEEL DIE

This section describes the die material and its different component used to prepare a rubber punch and FRP component.

4.4.1. Material

The material for the die chosen is mild steel as mild steel has high strength, good macinability, good thermal conductivity, high stiffness and good compressive strength. Thus the material is suitable to bear high compressive stresses developed during the curing of performs under pressure. The same die is also used to prepare rubber punch. Also it does not adhere to the cured rubber, therefore cured rubber punch could be easily taken out from the die. Moreover deformation of mild steel die is negligible.

4.4.2 Preparation of Die

The mild steel die is made of two parts (split type). Its assembly drawing is shown in Fig 4.3. Two parts of the die are symmetric to each other. To make the die a rectangular steel block is cut in to two pieces and their surfaces are polished to get smooth surface. Then two parts are tacked welded and machining is done on the welded assembly to

obtain the exact internal dimension of die. The inner surface of die is polished to obtain very smooth surface as the shape of rubber punch and preform depends on the surface finish of die. Four steel plates (125x25x10 mm) with a dowel hole (Φ8) and two M12 holes on each plate (shown in Fig 4.3) are welded. To sustain high pressure supporting webs are provided on the steel plates. Then the halves of the welded assembly are detached by breaking the welded points. Different allowances are taken into consideration to prepare actual size of FRP product.

As the component prepared is of only 1.5 mm thick, a single die set is used for preparing rubber punch as well as FRP components. However to prepare thicker component two die sets will be required, one for preparing the rubber punch and another one for preparing the component. In that case the internal dimension of the die to prepare rubber punch should be less and upset by thickness of component.

4.5 PREPARATION OF RUBBER PUNCH

Four types of rubber (natural, butyl, silicon and polybutadiene) are used to cast four different rubber punches. The rubber punch that is to cast have a same inner shape that of FRP component. The die used to cast the rubber punch is same that is used to prepare the component i.e. split steel die. This section describes design and fabrication of rubber punch and procedure to prepare rubber punch.

4.5.1 Design and fabrication of rubber punch.

The split steel die (Fig 4.3) and the steel cone are used for casting a rubber punch by pressing uncured rubber between split steel die and steel cone.

The material for steel cone chosen is mild steel. It consists of a circular base with a conical projection. The circular base has a dimension of Φ119.8 mm X 10 mm thickness. The conical portion has a diameter of 85 mm, 54° half cone angle and height of 75 mm. Two holes of diameter 3 mm each are provided on the circular base to

bleed off extra rubber material during curing of rubber punch. The whole assembly is shown in Fig 4.4.

4.5.2 Preparation of uncured rubber

The uncured rubber is prepared in "two roll mixing machine" shown in Fig 4.5. The ingredients mixed with different rubber are given in Table 4.1. Various chemicals mixed one at a time in the prescribed sequence of zinc oxide, steric acid, carbon black, sulphur and accelerator a temperature of 50°C. The roller gap is 0.5 mm during mixing of all the ingredients. Mixing time of half an hour is required to prepare uncured rubber.

Table 4.1 composition of ingredients in 100 gms of rubber

Rubbers (100 gms)	Zinc Oxide (in gms)	Steric acid (in gms)	Carbon Black (in gms)	Sulphur (in gms)	Accelerator (in gms)
Natural	5.0	2.5	50.0	2.0	2.0
Butyl	5.0	2.0	50.0	2.5	2.5
Silicon	5.0	2.5	50.0	2.0	2.0
Polybutadiene	5.0	2.0	50.0	2.5	2.5

4.5.3 Preparation of rubber punch

The split steel die is preheated before putting rubber in it. The preheating is done in a hydraulic press to a temperature of 150°C. After temperature is reached close to 150°C, uncured rubber is filled in the die and the steel cone is placed over it. A nut and bolt system is placed inside the rubber punch (Fig 4.6) for easy removal of the component from the rubber punch after curing of preform. The pressure and temperature applied on the die set varies from rubber to rubber as given in Table 4.2. The temperature and pressure is maintained for 30 minutes. The variations of pressure and temperature in different rubber are to get a constant hardness of 40 shores A. The cured rubber punch is

taken out by removing bolts on the metal plate of die. The photograph of the rubber punch is shown in Fig 4.7.

Table 4.2: Pressure and temperature cycle applied for preparation of rubber punch

Rubbers	Temperature (°C)	Pressure (MPa)
Natural	150	5.5
Butyl	160	5.0
Silicon	160	5.0
Polybutadiene	150	5.0

4.6 FABRICATION OF PRODUCT

This section describes the fabrication of FRP product (pump cap) using rubber molding technique. The FRP products are made using four varieties of rubber punches. These are (i) natural, (ii) butyl, (iii) silicon and (iv) polybutadiene rubber punch. Pump cap is prepared using above four verities of rubber to check the feasibility of different rubber punches. The toolings required for molding a pump cap are split steel die, rubber punch and steel cone. The photographs of split steel die, rubber punch and steel cone together are shown in Fig 4.8.

4.6.1 Material required

The pump cap is made using glass fiber fabric and epoxy resin. The specifications of glass fiber and epoxy resin are given in Tables 4.3 and 4.4 respectively.

Table 4.3 Specification of E-glass fiber

Parameters	Values
Density, Kg/m^3	2540
Tensile strength, MPa	2450
Elastic Modulus, GPa	72.40
Range of diameter, μm	3-20
Coefficient of thermal expansion 10^{-6} / °C	5.0

Table 4.4 Specification of the epoxy resin (at 23°C) in cured stage

Parameters	Values
Density, 10^6 Kg/m^3	1.2 -1.3
Tensile strength, MPa	55-130
Tensile Modulus, GPa	2.75-4.10
Water absorption, % in 24 hr.	0.08-0.15
Coefficient of thermal expansion 10^{-6} / °C	45-65

4.6.2 Procedure to fabricate FRP product using rubber molding technique

The procedure for preparing FRP product is as follows:

(1) Preparation of die and punch: The oil and grease present on the surface of the split steel die and the rubber punch are removed by using acetone. Then a thin layer of mold release agent, PVA is applied on the surface of the die to prevent adhesion of resin to steel die and easy removal of the FRP product from the metal die after curing. After applying a thin coating of release agent, it is kept at room temperature for an hour to form a thin layer of PVA on the surface of the steel die.

(2) Making of preform: A hand lay-up technique is used to prepare the preform (uncured form of product). The glass fabric is cut in right shape using a template 1 (Fig 4.9 (a)) from a woven glass fiber sheet. The template is designed in such way that the uncut portion covers cylindrical portion of the product and the cut portion covers the conical and base portion of the product. The conical and base of the product does not give continuous laying, so template 2 and template 3 (Figs 4.9(b) and 4.9(c)) are inserted in conical and base portion after every two layers of template 1. The matrix used to prepare the product is epoxy resin. The epoxy resin is mixed with 10% of hardener (HY951) to cure the resin. Total numbers of template 1, template 2 and template 3 used to prepare the preform are 4, 2, and 2 respectively. Using hand lay-up technique the glass fabric and the epoxy resin are placed on the rubber punch, then the rubber punch with preform is placed inside the split steel die and four bolts of the die set is bolted till the two portion of the split die touches each other. Then, steel cone is placed over the rubber punch.

(3) Curing of preform: The steel die and the rubber punch with preform are loaded in a hydraulic press. The technical specification of the hydraulic press is given in Table 4.5. The pressure cycle used in the hydraulic press is 0.4 MPa and the preform is allowed to cure at room temperature for 16 hours.

Table 4.5: Specification of the hydraulic press

Capacity	25 Ton
Platen size	460x460 (mm)
Piston area	18400 mm^2
Temperature range	Room temperature to 400 ^{o}C
Heater	Electric heater (both platen is controlled by separate temperature controller)

(4) Removal of product: The steel die is separated in to two parts by removing the bolts after curing of preform. To take out the product from the rubber punch, a M12 bolt is placed on the M12 nut inside the rubber punch and screwed slowly to take out the

product from the rubber punch. The purpose of placing the nut and a steel plate inside the rubber punch to take out the product easily with out hammering the rubber and the FRP product. The removal of product from the die and punch should be done very carefully i.e. the surface of the die, punch and the product should not be damaged. The product is then smoothened with the help of an emery paper.

4.7 PREPARATION OF TEST LAMINATES

The FRP specimens with various geometries are prepared for different mechanical testing to determine various mechanical properties. Five specimens are prepared from one laminate for testing of tensile strength, interlaminar fracture toughness and interlaminar shear strength. The methods of preparation include conventional method and rubber molding techniques (using natural, butyl, silicon and polybutadiene rubber sheet). This section describes the die and punch to prepare rubber sheets and procedure for specimen preparation.

4.7.1 Fabrication of steel die and punch to prepare rubber sheet

A set of die and punch is designed and fabricated to prepare rubber sheets with smooth surfaces. Mild steel is chosen as the die and punch material because of its high strength, good thermal conductivity and good machinabilty properties. The details of steel die and steel punch are shown in Figs 4. 10 (a) and (b) respectively. The die is made from a mild steel plate. The plate is machined to get dimension of 250 X 250X 17 mm. Across the periphery of the steel die metal strips of 3 mm thickness and 7 mm width are bolted (as shown as in Fig 10 (a)) to obtain 3 mm thick rubber sheet. Shift at two corners are provided for excess material to flow out. The punch used is a mild steel plate with dimension of 250 X 250 X 20 mm. The surfaces of die and punch are polished to get very smooth surface as the surface of rubber sheet depends on the surface finish of die and punch in contact with it during casting of rubber sheet.

4.7.2 Casting of rubber sheet

Using the steel die and punch (Figs 4.10 (a) and (b)) four varieties rubber sheets (natural, butyl, silicon and polybutadiene rubber sheets) are prepared. The width, length and the thickness of the rubber sheet are 212, 212 and 3 mm respectively. Uncured rubbers are prepared (Section 4.5.2) with different proportions of chemical ingredients in two roll mixing machine. Steel die and steel punch is preheated to 150 °C. Then 5% extra amount of uncured rubber than the required amount to prepare the rubber sheet is put in to the steel die and steel punch is placed over the steel die. The whole assembly is placed in a hydraulic press. The temperature and pressure cycle given in Table 4.2 are maintained for 30 minutes for different rubbers to cure the rubber and get the rubber sheet with specified geometry.

4.7.3 Procedure for specimen preparation

The specimens used in the present work are flat laminates prepared from glass fabric and epoxy resin using conventional and rubber molding techniques .The schematic diagrams of these processes are shown in Figs 4.11 and 4.12 respectively. The preform is prepared by hand lay-up technique. The stacking is carried out by placing warp of all layers in same direction. The epoxy resin is prepared by adding 10% of hardener (HY951) by weight. In conventional method, the preform along with mylar sheets are placed in between top and bottom metal platen of the hydraulic press where as in rubber molding technique, preform with mylar sheet on one face and rubber sheet on the other side are pressed in the hydraulic press. According to the requirement of thickness and volume fraction of fiber, different pressure cycles are applied. In the present work, all specimens have volume fraction of fiber in the range of 0.5 to 0.54 using a pressure of 0.4 MPa. The pressure is maintained for 16 hours at room temperature to cure the laminates. The cured laminate is then taken out from the press and cut to the required size with the help of diamond cutter.

4.8 TESTING OF FRP PRODUCT

A good composite product should have an optimum fiber to resin ratio. Volume fraction of fiber 40-55% and void content less than 5%. Two pieces of FRP pump cap from each rubber punch are made using rubber molding technique. A series of test like burn test, coin test and microstructure study are done to study the quality of product. This chapter describes above three quality tests of four varieties of pump cap prepared by using four varieties of rubber punches used in the present work. The photographs of tested products are given in Figs 4.13 and 4.14.

4.8.1 Burn test of FRP product

In burn test, volume fraction of the fiber, matrix and void contents are calculated. This test has been conducted for cylindrical, conical and flat part of the FRP pump cap. Procedure to conduct the burn test is as follows.

(i) A specimen of size 10X10 mm is cut from the product.

(ii) The specimen is cleaned and dried to remove unwanted material from the specimen, and then the specimen is weighted in air and water to determine the specific gravity of the composite materials.

(iii) It is kept in a furnace and the furnace temperature is gradually increased to 1000°C to burn out resin completely. It is hold at 1000°C for 20 minutes and furnace is switched off. The residue is taken out, when it is cooled to room temperature and its weight is taken, which gives the weight of the fiber.

From the specific gravity of the composite specimen, weight of the fiber, density of resin and density of fiber, one can find the volume fractions of fiber, matrix and void content in the specimen.

The following equations are used to determine volume fraction of fiber and void content in the composite specimen [Agarwal and Broutman, 1990].

Density of the composite specimen, ρ_c is given by

$$\rho_c = \frac{m_a \cdot \rho_w}{m_a - m_w} \tag{4.1}$$

where ρ_w, m_a and m_w are density of water, weight of specimen in air and weight of the specimen in water respectively. Weight of the fiber (m_f) and the matrix (m_m) are obtained after burning the specimen. Volume fraction of the fiber (V_f) and void content (V_v) are given by

$$V_f = \frac{\rho_c}{\rho_f} w_f \tag{4.2}$$

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \tag{4.3}$$

where, ρ_f, ρ_{ct} and ρ_{ce} are density of the fiber, theoretical and experimental density of the specimen respectively.

Theoretical density of composite is given by

$$\rho_{ct} = \frac{1}{\frac{w_f}{\rho_f} + \frac{w_m}{\rho_m}} \tag{4.4}$$

Five specimens are cut from each sections of pump cap, i.e. cylindrical, conical and flat surfaces and burn test is conducted to find out volume fraction of fiber and void contents. Burn test data for the products are given in Table 4.6

Table 4.6: Burn test data of FRP products.

Product name	Part name	Volume Fraction (%)		Standard deviation of volume fraction	
		Fiber	Void	Fiber	void
NR -1	Cylindrical	45.4	3.0	0.75	0.63
	Conical	44.6	2.7	0.63	0.64
	Flat	45.3	2.9	0.71	0.68
BU -1	Cylindrical	46.3	2.9	0.54	0.54
	Conical	44.9	2.5	0.63	0.53
	Flat	45.3	2.6	0.69	0.61
SI -1	Cylindrical	45.2	2.6	0.58	0.53
	Conical	44.8	2.4	0.54	0.64
	Flat	45.5	2.5	0.62	0.59
PB-1	Cylindrical	45.7	2.9	0.66	0.66
	Conical	45.3	2.6	0.73	0.61
	Flat	47.1	2.7	0.65	0.68

NR-1 = Natural rubber BU-1 = Butyl rubber,

SI-1 = Silicon rubber PB-1 = Polybutadiene rubber

The burn test data (Table 4.6) shows void contents of components are within reasonable range of 3.0 % and small variation of volume fraction of fiber with in the component. Standard deviation of volume fraction of fiber and void content are less than 0.1. Photograph of NR-1 and BU-1 is shown in Fig 4.13 and photograph of SI-1 and PB-1 is shown in Fig 4.14.

4.8.2 Coin Test

This test gives an idea of delamination in the FRP products. In this test, sound quality is checked while tapping a coin on the FRP product. If sound is like metal i.e. of high

frequency, it ensures good quality of product. Otherwise delamination or high void content may present in the product.

The coin test in the FRP product (pump cap) is found to be satisfactory in all the three parts (cylindrical, conical, and flat surface). However the sound in conical and flat surface is different than that of cylindrical surface, as in conical and flat surface continuous fiber fabric is not used. But all three surfaces give a metallic sound, which indicates that delamination in the FRP product, is absent.

4.8.3 Electron microscopy

Study of wetting characteristics and fiber-matrix interaction are done by JSM-840, scanning electron microscope, JEOL JAPAN. The specimen of size 10X10 mm is cut from cylindrical portion of pump cap and edge having cross section of fibers are smoothened by a waterproof emery paper. Then, the edge is polished by a powder, 0.3 micron alpha alumina, manufactured by Buehler Micropolish, USA. Scanning Electron microscope studies of the products are shown in the Figs. 4.15 to 4.18. These figures show good interaction between fiber and matrix.

4.9 COMPARISON OF FRP PRODUCTS MADE FROM DIFFERENT RUBBER PUNCHES

A comparison is made between the products made by rubber molding technique (using natural, butyl, silicon and polybutadiene rubber punch). Table 4.6 shows the fiber volume fractions and void contents of cylindrical, conical and flat surfaces of the FRP product (pump cap). It is clear from burn test that the void content in all three portions of FRP component with in the range of 3.0% for all methods. The difference in volume fraction of fiber and void contents in four varieties of product (using four varieties of rubber punches) has very small. Uniformity is maintained in all parts as the volume fractions of fiber are in agreement with all parts of the product. Coin test for all four types of product gives sounds like metal ensuring good quality of product with out

delaminations. Finally, Electron microscopy of all types of product shows good interaction between fiber and matrix. This difference can be neglected as the process of making preform is done by hand lay-up technique. Thus the FRP products prepared from all four varieties of rubber punch are of similar in nature and gives better uniformity through out surface.

4.10 CLOSURE

The FRP component fabricated using rubber molding technique is a pump cap. This technique is similar to matching die set, where die is made from mild steel and punch from rubber material. A split steel die and steel punch are designed and fabricated to prepare the rubber punch. Again with the help of split steel die, steel punch and rubber punch, the FRP components are prepared. In designing the split steel die, a special care has been taken to ensure easy removal of rubber punch and FRP product. Different tests like burn test, coin test and microstructure study are performed to check the product qualities like volume fraction fiber, void contents, possibility of delamination and interaction between fiber and matrix. The comparison of four varieties of product (using natural, butyl, silicon and polybutadiene rubber punch) has been done. It shows that there is not much difference in fiber volume faction and void contents in FRP component. All FRP products prepared from four verity of rubber punch are of similar in nature and gives better product with uniformity through out the surface due to uniform pressure distribution during preparation of FRP.

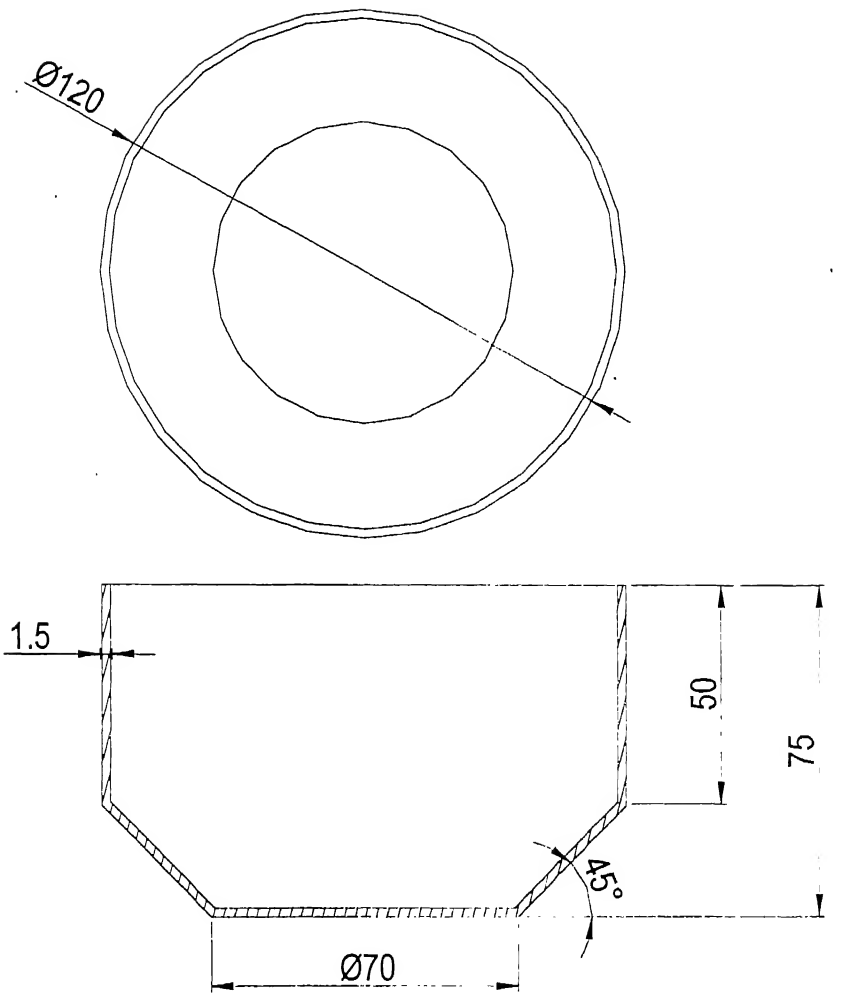


Fig. 4.1: Schematic diagram of pump cap

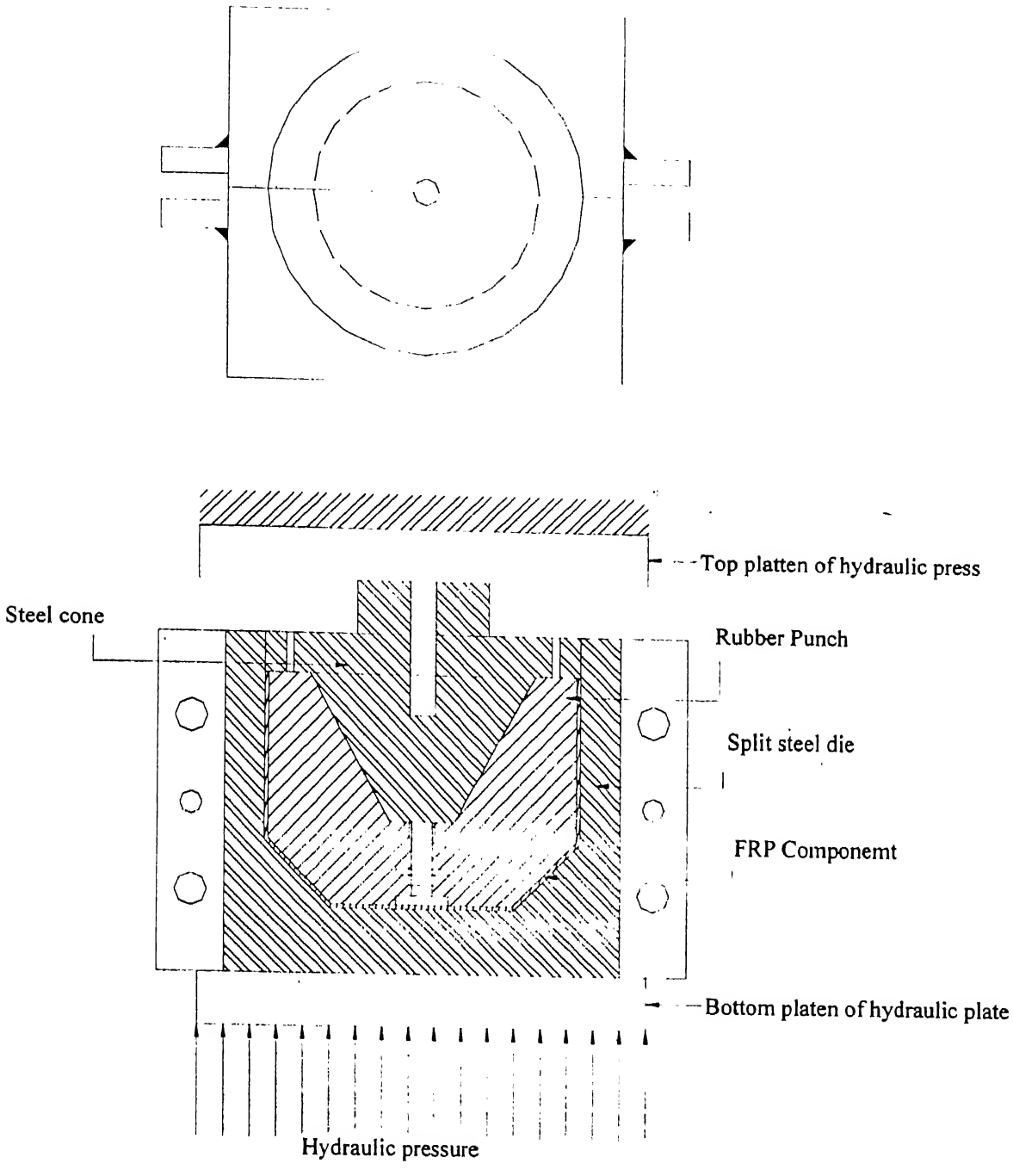


Fig 4.2: A schematic diagram of rubber molding technique

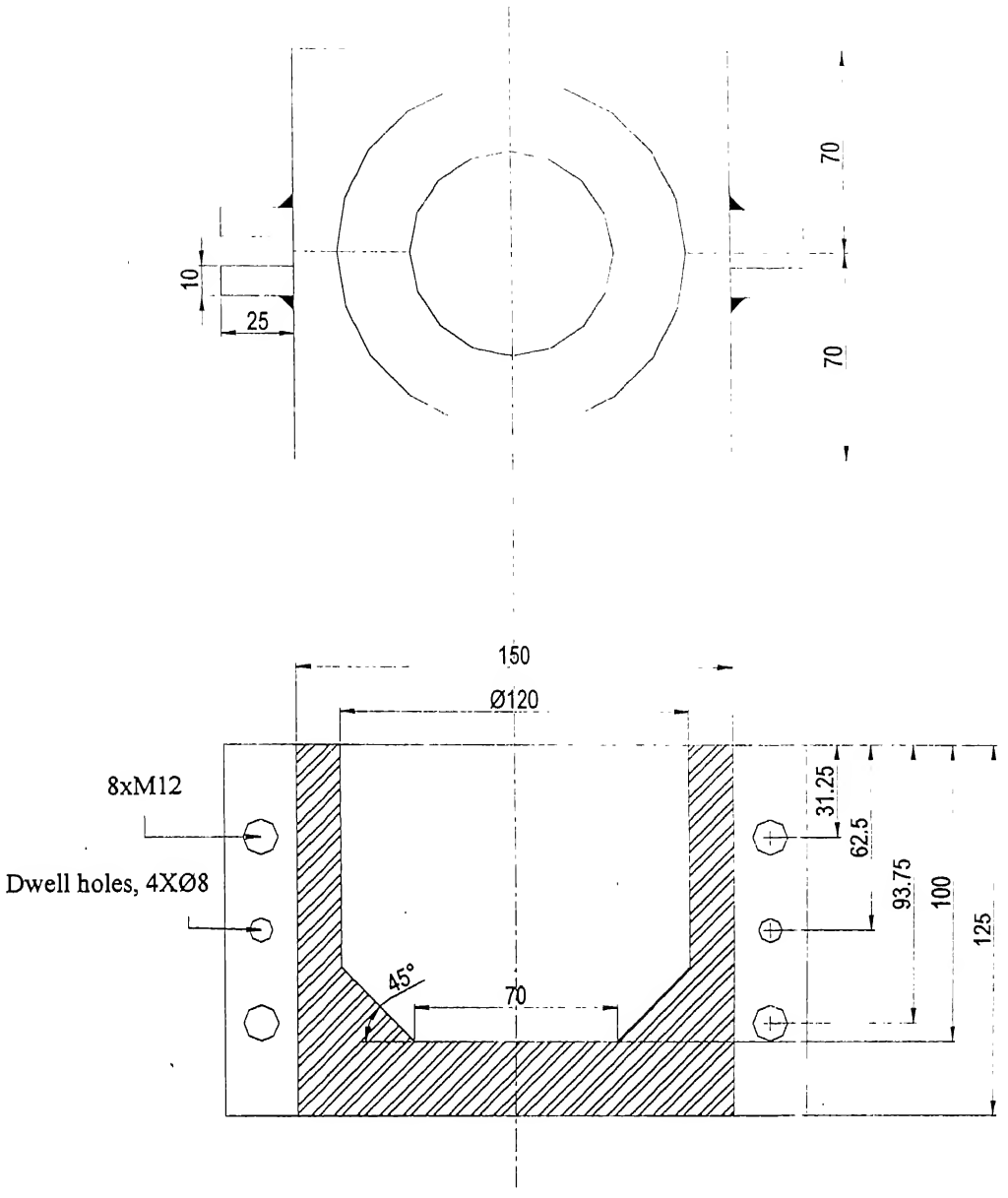


Fig 4.3: Split steel die

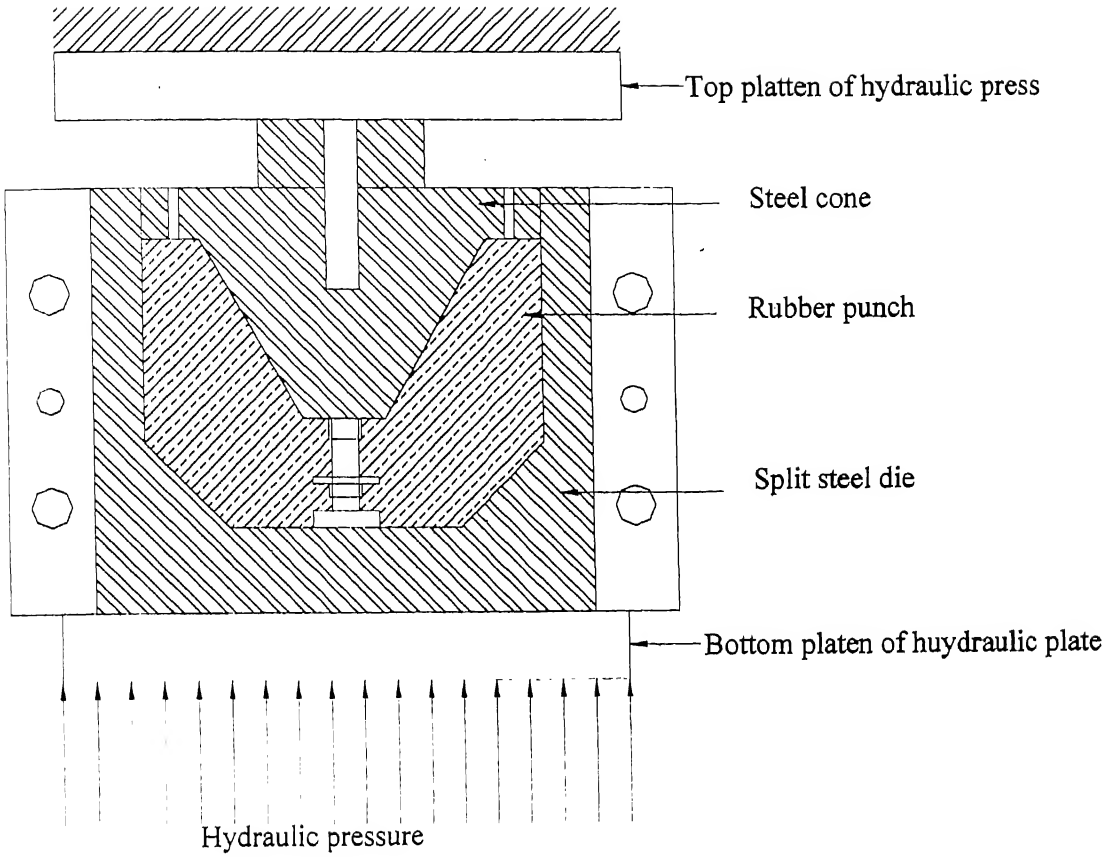


Fig 4.4: Schematic diagram for casting of rubber punch

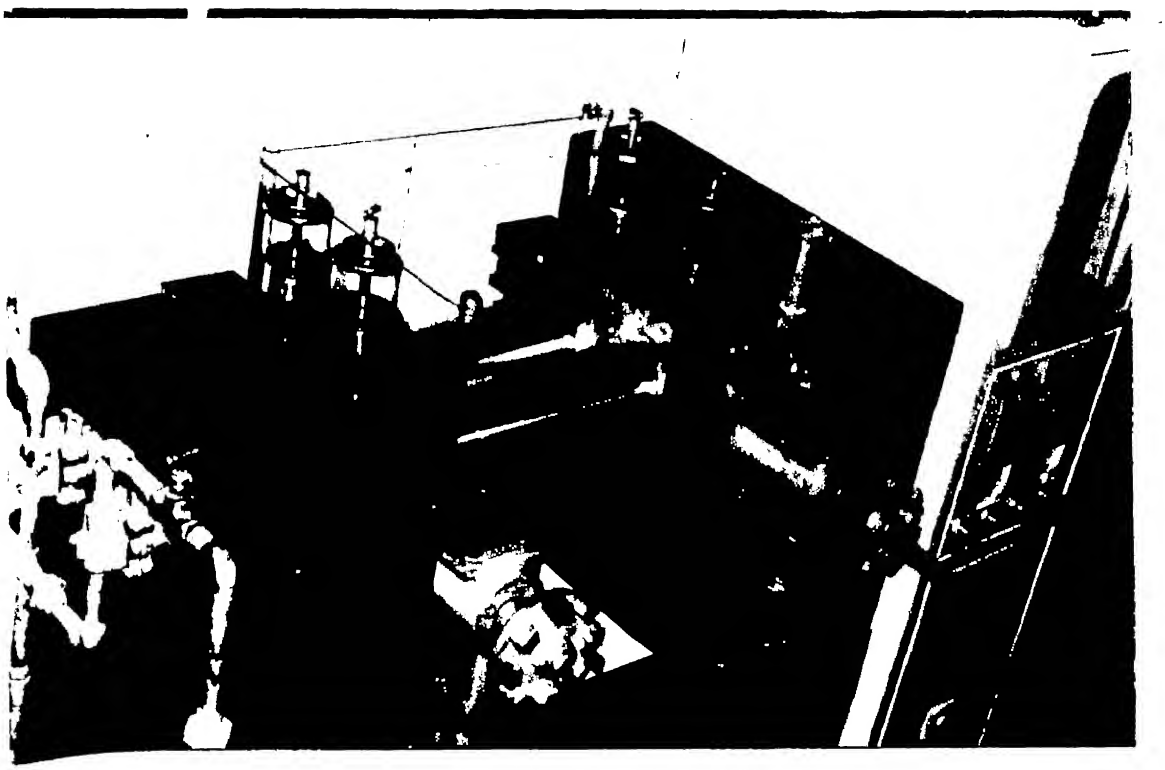


Fig 4.5: Photograph of two roll mixing machine

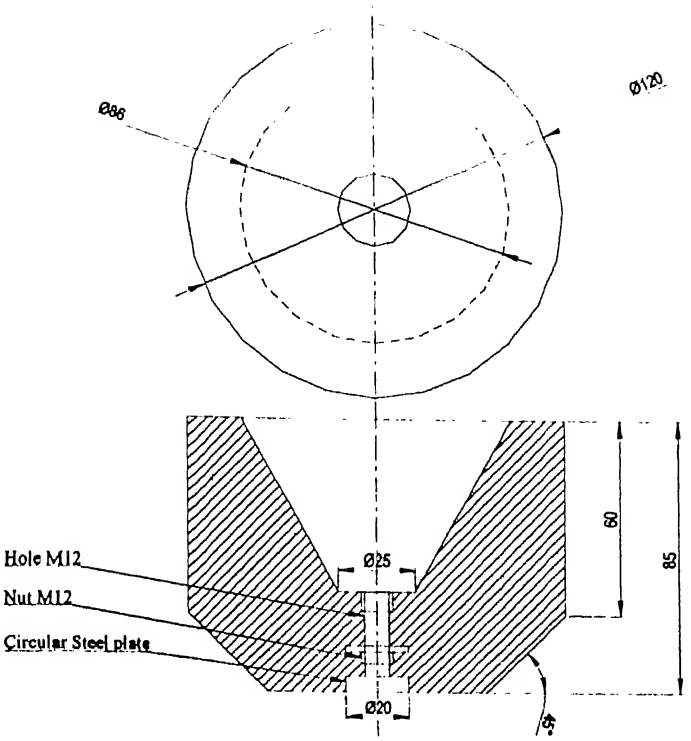
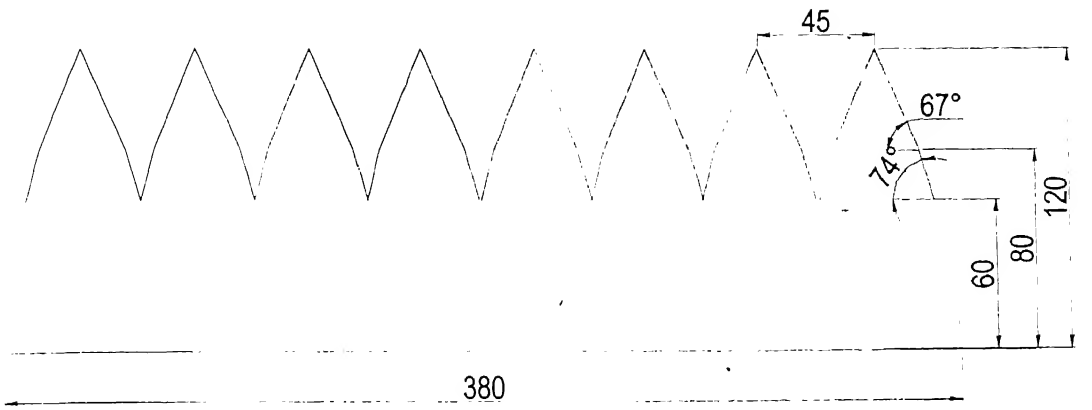


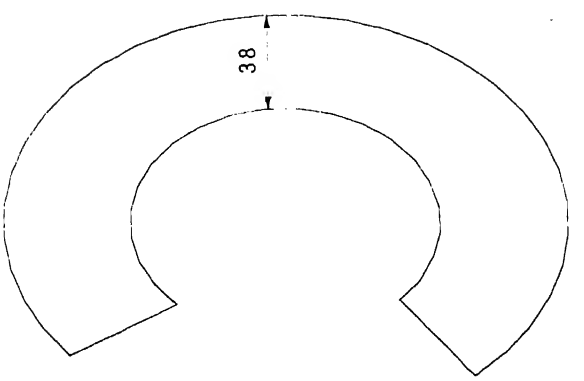
Fig: 4.6: Schematic diagram of rubber punch

Fig. 4.7: Photograph of rubber punch

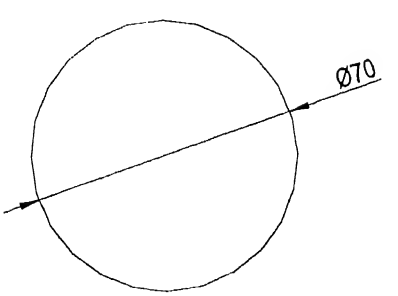
Fig: 4.8: Split steel die with rubber punch and steel cone



(a)

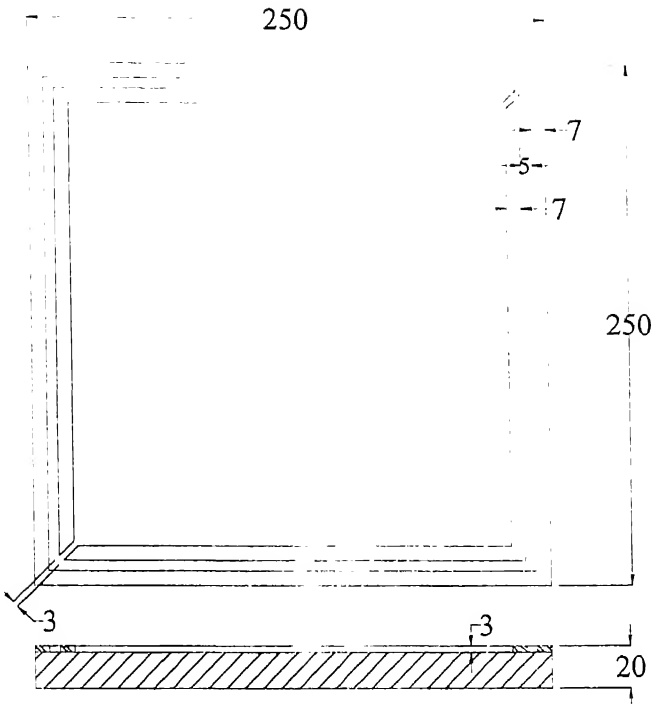


(b)

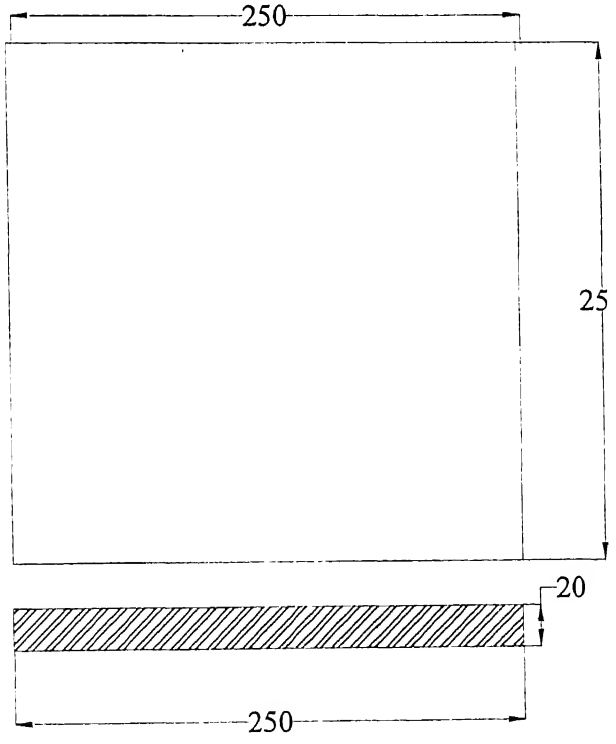


(c)

Fig. 4.9: (a) Template 1, (b) Template 2, (c) Template 3



(a)



(b)

Figs 4.10: (a) Steel die for rubber sheet preparation

(b) Steel punch for rubber sheet preparation

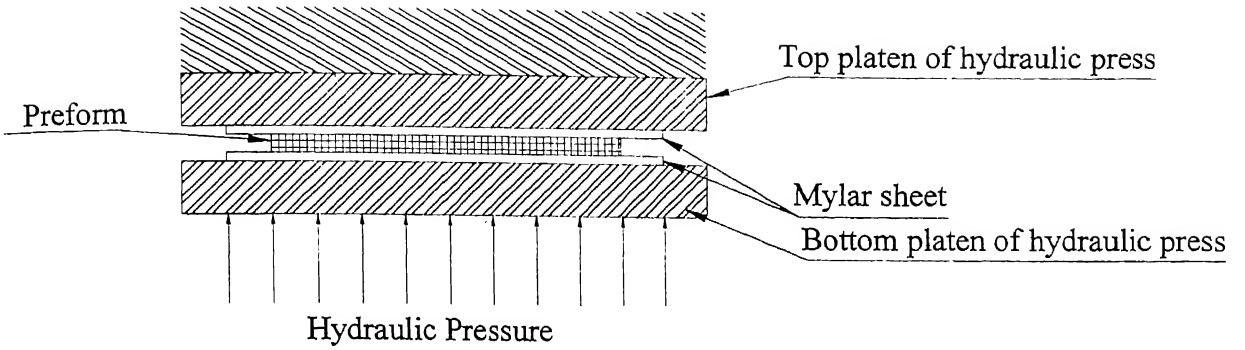


Fig.4.11: Conventional method of preparing composite laminate.

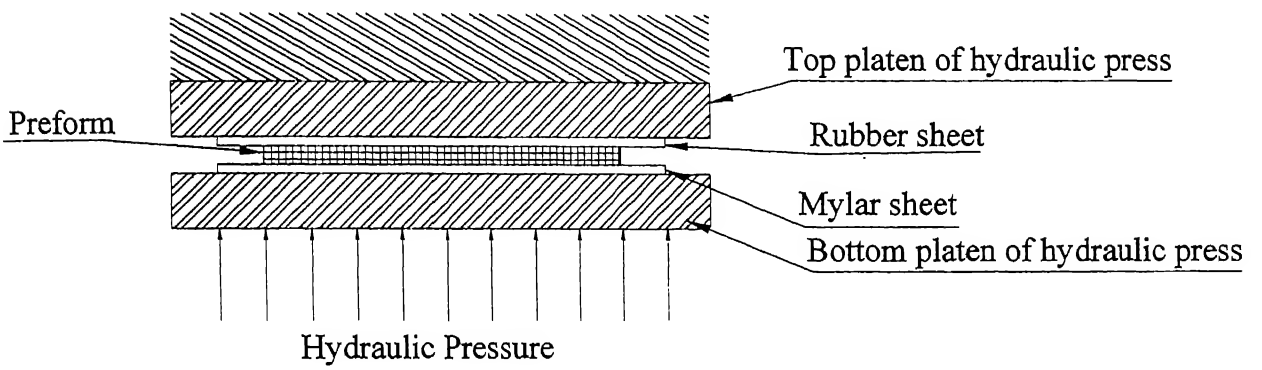


Fig. 4.12: Rubber molding method of preparing composite laminate.

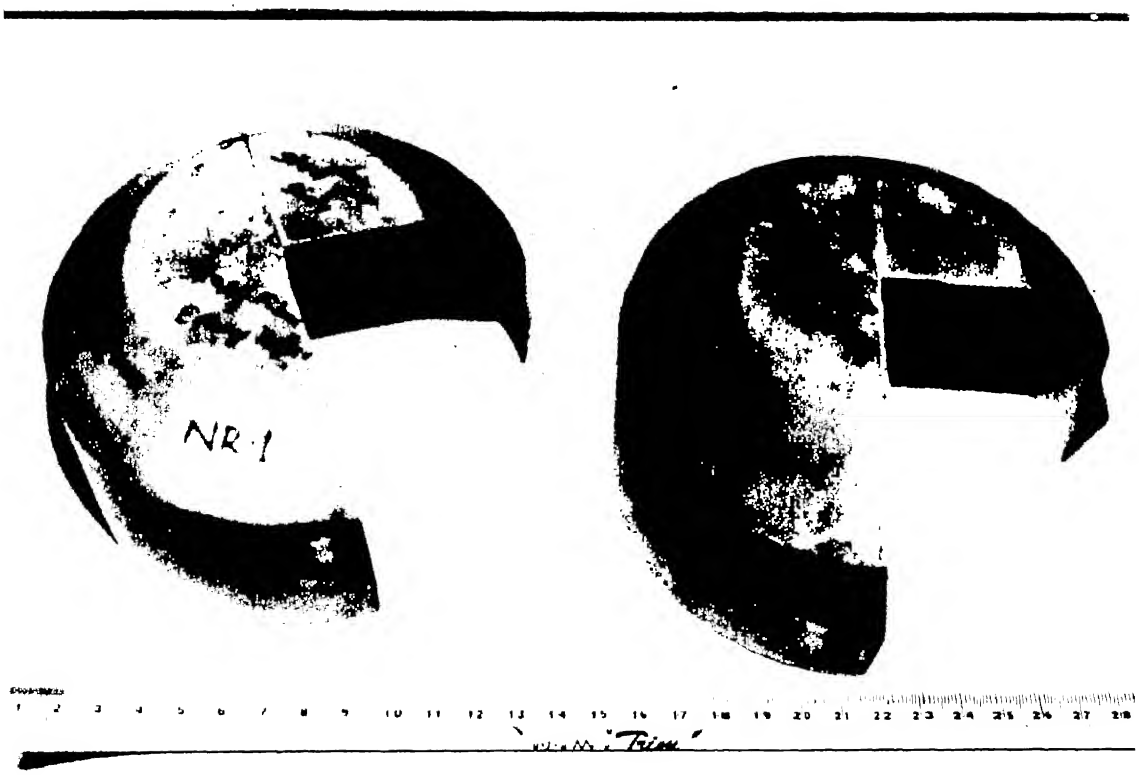


Fig. 4.13: Photograph of NR-1 and BU1

(NR-1: Made by using natural rubber, BU-1: Made by using butyl rubber)

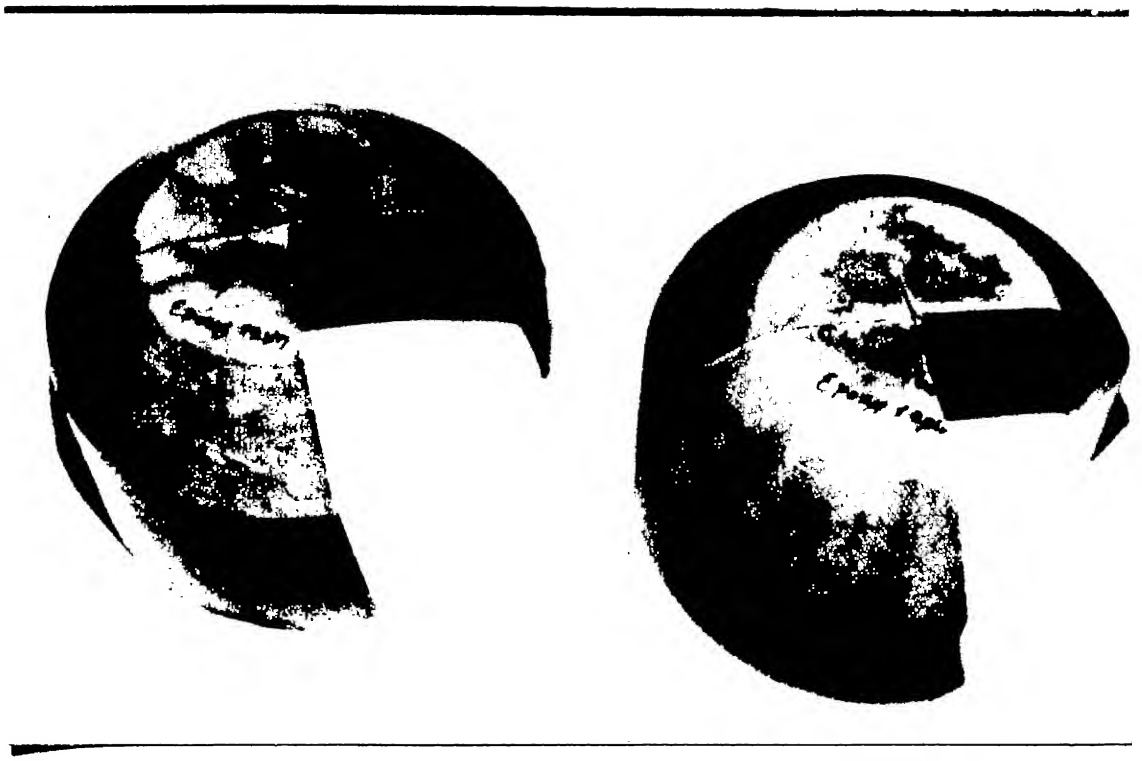


Fig. 4.14: Photograph of SI-1 and PB-1

(SI-1: Made by using silicon rubber, PB-1: Made by using polybutadiene rubber)

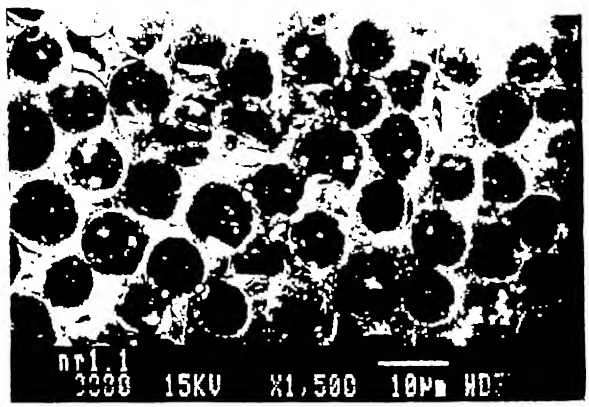


Fig. 4.15: Electron microscopy of NR-1 (Cylindrical part)

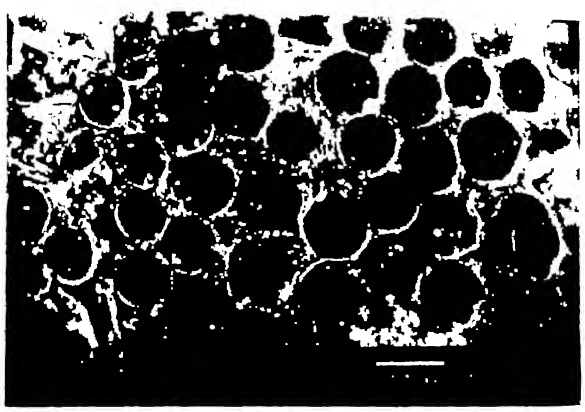


Fig. 4.16: Electron microscopy of BU-1 (Cylindrical part)

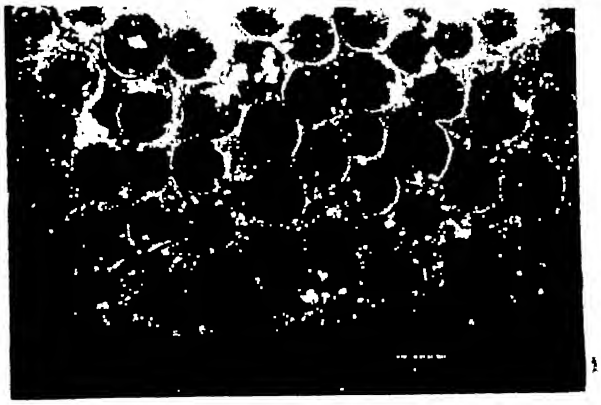


Fig. 4.17: Electron microscopy of SI-1 (Cylindrical part)

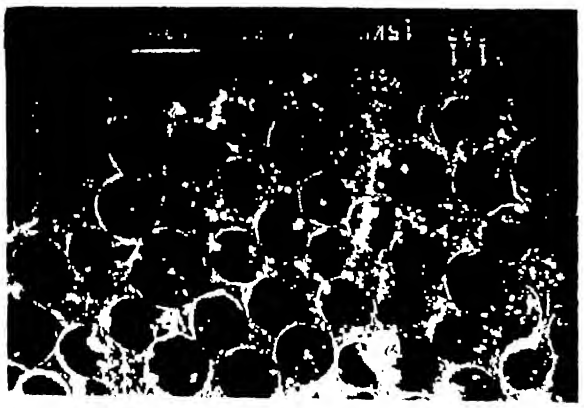


Fig.4.18: Electron microscopy of PB-1 (Cylindrical part)

CHAPTER-5
CHARACTERIZATION OF MATERIAL THROUGH INTERLMINAR FRACTURE TOUGHNESS TEST

5.1 INTRODUCTION

The aerospace, automobile and other industries used fibre reinforced composite materials since last four decades. But the biggest drawback, which has been noticed, is its low resistance to delamination. The delamination in laminates not only leads to complete fracture but also decreases its stiffness, which is a very important design parameter for designers. In the present scenario, it is a challenge for researchers to reduce this delaminating behaviour of composites in order to increase its life and load bearing capacity. The parameter "Energy Release Rate" suits for studying the crack due to delamination in composites. Because the crack plane is well defined and the material remains elastic in the vicinity of the crack tip except in the very thin layer of the interface.

This chapter introduce the concept of critical energy release rate (G_{Ic}) and to determine it using double cantilever beam (DCB) specimen using compliance method. The objective of present investigation is to determine the critical energy release rate of DCB specimen made by the conventional method and rubber molding technique. Again in rubber molding technique specimens are prepared using natural, butyl, and silicon and polybutadiene rubber sheets.

5.2 DETERMINATION OF INTERLAMINAR FRACTURE TOUGHNESS: MODE I

Mode I and mode II are the main failure modes by which interlaminar failure occurs. In mode I (opening mode), the applying load normal to crack plane as shown in Fig 5.1a, produces failure. But in mode II, in plane load causes failure (Fig 5.1b). Mode I is more

prevalent in fracture with well developed testing procedure. A number of reports are available in the literature for measurement of mode I interlaminar fracture toughness for composite materials. The DCB type configuration is one of the most common specimen geometry. This section deals the experimental method for determining the mode I fracture toughness in delamination with necessary theory. The critical energy release rate (G_{Ic}) is chosen as suitable parameter to characterize the fracture toughness.

5.2.1 Expression for energy release rate

Two important parameters are involved in any crack growing process. One is the energy release rate and other is crack resistance. There is always an energy release from the body when a crack moves and at the same time some amount of energy is required for the formation of new surfaces. The energy release is measured by a parameter, energy release rate, G. The energy release rate is defined as energy release for a crack to grow by unit area. Also the energy required to form new crack surfaces is accounted by a parameter, crack resistance, R. The crack resistance is defined as the energy required for a crack to grow by unit area.

Mathematical formulation for energy release rate is carried out by invoking the conservation of energy. Consider a body having a crack of length 'a' and if the crack grows by Δa then the incremental increase in the crack area is ΔA. For an incremental external work, ΔW_{ext}, by the external forces, strain energy balance is given by

$$G \cdot \Delta A = \Delta W_{ext} - \Delta U \tag{5.1}$$

where 'A' is the area of the crack. Dividing the Eq. 5.1 by ΔA and taking the limit $\Delta A \rightarrow 0$, the following equation is obtained

$$G = - \frac{d(U - W_{ext})}{dA} \tag{5.2}$$

But $(U - W_{ext})$ is commonly known as potential energy Π. Therefore, the above equation is written as

$$G = - \frac{d\Pi}{dA}. \tag{5.3}$$

Now consider the general case of a body with a crack and load P acting as shown in Fig 5.2. The displacement, u of the points at which load is applied can be written as

$$u = CP \tag{5.4}$$

where C is compliance.

Strain energy is then expressed as

$$U=\frac{1}{2}Pu \quad =\frac{1}{2}C.P^2 \tag{5.5}$$

Differentiating, one obtains

$$dU=CPdP+\frac{P^2}{2}dC. \tag{5.6}$$

External work done is given by

$$dW_{ext}=Pdu \tag{5.7}$$

Differentiating Eq. 5.4 and substituting in Eq. 5.7, one obtains

$$dW_{ext}=PCdP+P^2dC \tag{5.8}$$

Substituting from Eqs. 5.6 and 5.8 into Eq. 5.2 the following equation is obtained

$$G = \frac{P^2}{2}.\frac{dC}{dA}. \tag{5.9}$$

For a special case of DCB specimen of thickness 'b', $dA=b.da$ and then

$$G=\frac{P^2}{2b}\frac{dC}{da}. \tag{5.10}$$

When a body with a crack is loaded, the crack propagation begins at a specified load. Depending on the initial crack length, the crack may be stable or unstable.

The necessary conditions for the propagation of crack are

$$G \geq R \tag{5.11}$$

$$dG/da \geq dR/da \tag{5.12}$$

where R is crack resistance.

The value of G which satisfies these two conditions becomes a material property and is known as critical energy release rate, G_{Ic}.

5.2.2 Experimental method

There are several test methods to determine the Energy Release Rate [Kumar, 1999]. These are given below:

- Area method with DCB specimen
- Compliance method with DCB specimen
- Angle method with DCB specimen
- Embedded Crack Plate (ECP) test method

In the present work, compliance method with double cantilever beam (DCB) specimen is used. The compliance method using double cantilever beam (DCB) specimen has been widely used for measurement of interlaminar fracture toughness in mode I. The deflection of cantilever beam, δ caused by load P applied at the free end is given as

$$\delta = \frac{PL^3}{3EI} \tag{5.13}$$

where, L, E and I are length of cantilever arm, modulus of elasticity of material, and moment of inertia of beam cross section respectively.

For DCB specimen under a load P as shown in Fig 5.2, the cracked portion behaves as two independent cantilevers of length 'a'. Therefore, the relative displacement of the load is

$$u = 2\frac{Pa^3}{3EI} \tag{5.14}$$

and the compliance C is given by

$$C = \frac{u}{P} = \frac{2a^3}{3EI}. \tag{5.15}$$

Differentiating Eq. 5.15 with respect to 'a' and substituting in Eq.(5.10) one obtains

$$G_I = \frac{1}{b}\frac{P^2a^2}{EI} \tag{5.16}$$

The material property E and geometric property I are needed to calculate G_I using above Eq. 5.16. But it is possible to calculate G_I without determining E and I through separate tests and measurements.

Eq. (5.15) can be written as

$$C = A_1 a^3 \tag{5.17}$$

where,

$$A_1 = \frac{2}{3EI} \tag{5.18}$$

Similarly, Eq.(5.16) can also be written as

$$P = \frac{\sqrt{G_I EIb}}{a} = \frac{A_2}{a} \tag{5.19}$$

where

$$A_2 = \sqrt{G_I EIb} \tag{5.20}$$

By eliminating flexural rigidity, EI from Eqs. (5.18) and (5.20), one obtains

$$G_I = \frac{3}{2}\frac{A_1 A_2^{\,2}}{b} \tag{5.21}$$

The G_I becomes material property, G_{Ic} when the applied load is critical load. Therefore, G_{Ic} can be evaluated by knowing A_1 and A_2. It is worth noting that A_1 and A_2 are constants for a given specimen and can be evaluated from experimental data obtained from DCB test and using an approximate data reduction technique.

Data reduction technique to determine G_{Ic}

To determine G_{Ic}, DCB specimen is pulled in a tensile test machine on the displacement control with low pulling speed and the crack is allowed to grow by a small distance. The machine is stopped for some time till the crack tip become stationary. The loading curve cycle gives value of crack length, critical load and compliance. The distance from the loading point to the crack tip marked along the specimen is measured as the crack length. The critical load is taken as the loading point, where there is significant change in slope of the loading curve. And compliance as the slope of the loading curve. In order to get compliance for increased crack length, the load on the specimen is decreased by moving tensile machine in the opposite direction. The machine is stopped at zero load. The crack length, compliance and critical load are determined by repeating the process. The crack length for each loading is denoted as $a_0, a_1 \ldots\ldots\ldots a_n$. The

critical load for the crack initiation for each crack length is obtained from the graph (Fig 5.3) as P_{c0}, P_{c1}, ... P_{cn}. For each crack length compliance of the specimen is calculated from slope of loading curve and denoted as C_0, C_1,..... C_n.

Data analysis for finding G_{Ic} requires fitting a straight line of a known slope to experimentally recorded data [Kumar,1999]. The line is $y = mx + c$ for which slope m is known and c is to be determined. The line is best fitted so as to minimize sum of square of the distance from the data points to the line. Consider a point (x_i, y_i) and the distance of the point from the line d_i, the sum of D of square of the distance for n points is

$$D = \sum_{i=1}^{n} d_i^2 = \sum \frac{(mx_i - y + c)^2}{m^2 + 1} \tag{5.22}$$

For minimum D, $$\frac{dD}{dc} = 0 \tag{5.23}$$

leading to $$c = \frac{(-\sum mx_i + \sum y_i)}{n} \tag{5.24}$$

Taking log of Eq (5.17) one obtains

$$\log C = 3 \log a + \log A_1 \tag{5.25}$$

Equation 5.25 can be represented as $y = mx + c$ with m=3, so one obtains

$$\log A_1 = \frac{1}{n}\left[-3\sum \log a + \sum \log C\right] \tag{5.26}$$

Similarly from Eq. (5.19) one can have

$$\log P = - \log a + \log A_2 \tag{5.27}$$

with slope m = -1, one obtains

$$\log A_1 = \frac{1}{n}\left[\sum \log a + \sum \log P\right] \tag{5.28}$$

Now knowing A_1 and A_2 from Eqs 5.26 and 5.28 respectively, the value of G_{Ic} can be determined from Eq. (5.21).

5.3 FABRICATION OF DCB SPECIMEN

The DCB specimen is prepared using conventional method as well as by rubber molding technique. 12 layers of bi-directional glass fiber are used to prepare the laminate. The matrix used is epoxy resin. The thickness of laminate is 4 mm obtained under a pressure of 0.5 MPa. The specifications of glass fiber fabric and epoxy resin are already described in Chapter 4.

A precarck is introduced in the laminate by inserting a thin sheet of BOPP (biaxially oriented polypropylene) film at the mid plane of the laminate during the stacking of glass fiber fabric. The BOPP film is kept at one end in the midplane during the fabrication of the plate as shown in Fig 5.4. For conventional method, the laminate is pressed by keeping it in between two mylar sheets in the hydraulic press and for rubber molding process the laminate is kept in between a mylar sheet and a rubber sheet. Four types of rubber sheets (natural, butyl, silicon and polybutadiene rubber) are used to prepare laminates. The laminate is kept for 16 hours at a temperature of 25°C and pressure of 0.5 MPa to get cure.

After the laminate is prepared, it is cut to the specified size of the specimen for testing. The dimensions of the specimen are given below and also shown in (Fig 5.2).

Length, $L = 200$ mm

Precrack length, $a_0 = 40$ mm

Width, $b = 30$ mm

Thickness, $2h = 4$ mm

The ends of the specimen are prepared in such a way that they can be fixed to the loading fixture [Fig 5.5]. The loading fixture consists of top and bottom rectangular tabs which are fitted to the specimen with the help of bolts. The tabs are tightened by the bolt from opposite side of the tabs such that the bolts only touches the respective tabs not other one. The specimen is painted with white color on the both side along thickness to mark the crack tip accurately during the experiment. A thin strip from a graph sheet is bonded on the top of the specimen to measure the crack length.

5.4 EXPERIMENTAL

The test set up in Instron machine shown in Fig 5.5 is used for measurement of mode I interlaminar facture toughness. The specimens are fixed with rectangular tabs shown in Fig 5.5. The top and bottom rectangular tabs are hold by the jaw of the Instron machine and properly aligned. The specimen is tested in the tensile mode with a crosshead speed of 1 mm/min. The load cell with maximum capacity of 100 kN is used and the load range is set to 0.2 kN. The chart speed set to 10mm/min. A real time display of load vs. deflection is obtained. During loading, when a sudden change in the slope of the loading curve is observed. The machine is then stopped and waits for some time for the crack to self arrest. Then the exact crack tip portion is marked on the specimen on both side of the specimen. To locate exact point of crack tip a magnifying lens is used. The specimen is unloaded and the machine is stopped at zero load. The machine is reloaded to get the similar plot for the next crack length. The experiment is repeated for 5-6 times. The Fig 5.3 shows critical load $P_{c0}, P_{c1}, \ldots\ldots P_{cn}$ for corresponding crack length of $a_0, a_1, \ldots\ldots a_n$. The first cycle is excluded from the calculation as cantilevers are partially bonded to the BOPP precarck and they are not free to move. From each method five specimens are prepared and tested.

5.5 RESULTS AND DISCUSSION

In the load-displacement curve (Fig 5.3) a hysterisis loop is found in each cycle. The first cycle is excluded from the calculation of G_{Ic}. The first loading cycle in all experiments observed to be non-linear because of some disturbances like BOPP sheets placed in the mid plane of the specimen sticks to both cantilever or during cutting the specimen to the specified size some filler materials sticks to the precrack surface. When the machine is switched off after the crack propagation, one can observed the drop of load with time, which indicates that the crack still grows after stopping the machine till self arrest. When the specimen is unloaded to zero load, a small permanent deflection is

observed. However, the permanent deflection at zero load is much smaller than the displacement in the loaded condition and its effect is neglected in the analysis.

The results of the tests are tabulated in Tables 5.1 to 5.5. The average value of G_{Ic} for the specimen made by conventional method with volume fraction of fiber 51% is 210±19 J/m^2. The average value of G_{Ic} for the specimen made by rubber molding technique using natural, butyl, silicon and polybutadiene rubber sheets are 166±16 J/m^2, 208±15 J/m^2, , 205±14 J/m^2 and 175±3 J/m^2 respectively. The fiber volume fraction of the specimen made by rubber molding technique using natural, butyl, polybutadiene and silicon rubber sheets are 54%, 53%, 54% and 52% respectively. The specimen prepared by rubber molding techniques using butyl sheet and silicon sheet have equivalent interlaminar facture toughness in fact marginally lower compared to the specimen prepared by conventional method. But the variation is with in the experimental error band. However the specimen prepared by using natural rubber and polybutadiene found to have significantly lower by 21% and 17% respectively compared to specimen prepared by conventional method.

5.6 CLOSURE

The parameter, critical energy release rate (G_{Ic}) is used to determine the interlaminar fracture toughness. Specimens for the measurement of interlaminar fracture toughness are prepared using both the techniques conventional and rubber molding techniques. Again in rubber molding technique four varieties of rubber sheets (natural, butyl, silicon, and polybutadiene rubber) are used to prepare the specimen. It is clear from the experimental results that the laminates prepared by using butyl and silicon rubber sheet have equivalent interlaminar fracture toughness value compared with the specimen prepared by the conventional method. But the specimen prepared by using natural rubber and polybutadiene found to have significantly lower interlaminar fracture toughness by 21% and 17% respectively compared to specimen prepared by conventional method.

Table 5.1: Interlaminar fracture toughness of the composite materials prepared by conventional method (V_f=0.51)

DCB No.	Width, *b* (mm)	Thickness of specimen, 2*h* (mm)	Range of crack length, *a* (mm)	No of cycles	G_{1c} (J/m^2)	Average G_{1c} (J/m^2)
1	30	4.0	42.0-64.5	5	234	210±19
2	30	4.0	45.0-62.5	5	190	
3	30	4.0	41.5-64.0	5	200	
4	30	4.0	42.5-63.0	5	200	
5	30	4.0	40.0-71.5	5	227	
Standard deviation σ = 19 J/m^2						

Table 5.2: Interlaminar fracture toughness of the composite materials prepared by rubber molding technique (using natural rubber sheet) (V_f=0.54)

DCB No.	Width, *b* (mm)	Thickness of specimen, 2*h* (mm)	Range of crack length, *a* (mm)	No of cycles	G_{1c} (J/m^2)	Average G_{1c} (J/m^2)
1	30	4.0	41.0-61.0	5	170	166±16
2	30	4.0	42.0-69.0	5	161	
3	30	4.0	45.5-62.0	5	147	
4	30	4.0	44.0-58.0	5	191	
5	30	4.0	46.0-59.5	5	161	
Standard deviation σ = 16 J/m^2						

Table 5.3: Interlaminar fracture toughness of the composite materials prepared by rubber molding technique (using butyl rubber sheet) (V_f=0.53)

DCB No.	Width, *b* (mm)	Thickness of specimen, 2*h* (mm)	Range of crack length, *a* (mm)	No of cycles	G_{1c} (J/m^2)	Average G_{1c} (J/m^2)
1	30	4.0	37.0-60.0	5	218	208±15
2	30	4.0	36.0-64.5	5	225	
3	30	4.0	39.0-58.0	5	191	
4	30	4.0	41.0-58.5	5	195	
5	30	4.0	41.5-55.0	5	211	
Standard deviation σ = 15 J/m^2						

Table 5.5: Interlaminar fracture toughness of the composite materials prepared by rubber molding technique (using silicon rubber sheet) (V_f=0.54)

DCB No.	Width, *b* (mm)	Thickness of specimen, 2*h* (mm)	Range of crack length, *a* (mm)	No of cycles	G_{1c} (J/m^2)	Average G_{1c} (J/m^2)
1	30	4.0	40.5-60.5	5	189	205±14
2	30	4.0	43.5-63.0	5	192	
3	30	4.0	41.0-63.0	5	210	
4	30	4.0	43.5-62.0	5	216	
5	30	4.0	42.5-63.0	5	217	
Standard deviation σ = 14 J/m^2						

Table 5.4: Interlaminar fracture toughness of the composite materials prepared by rubber molding technique (using polybutadiene rubber sheet) (V_f=0.52)

DCB No.	Width, *b* (mm)	Thickness of specimen, 2*h* (mm)	Range of crack length, *a* (mm)	No of cycles	G_{1c} (J/m^2)	Average G_{1c} (J/m^2)
1	30	4.0	43.5-63.0	5	171	
2	30	4.0	43.0-62.5	5	178	
3	30	4.0	43.0-63.0	5	174	175±3
4	30	4.0	43.5-60.5	5	178	
5	30	4.0	42.5-60.0	5	173	
Standard deviation σ = 3 J/m^2						

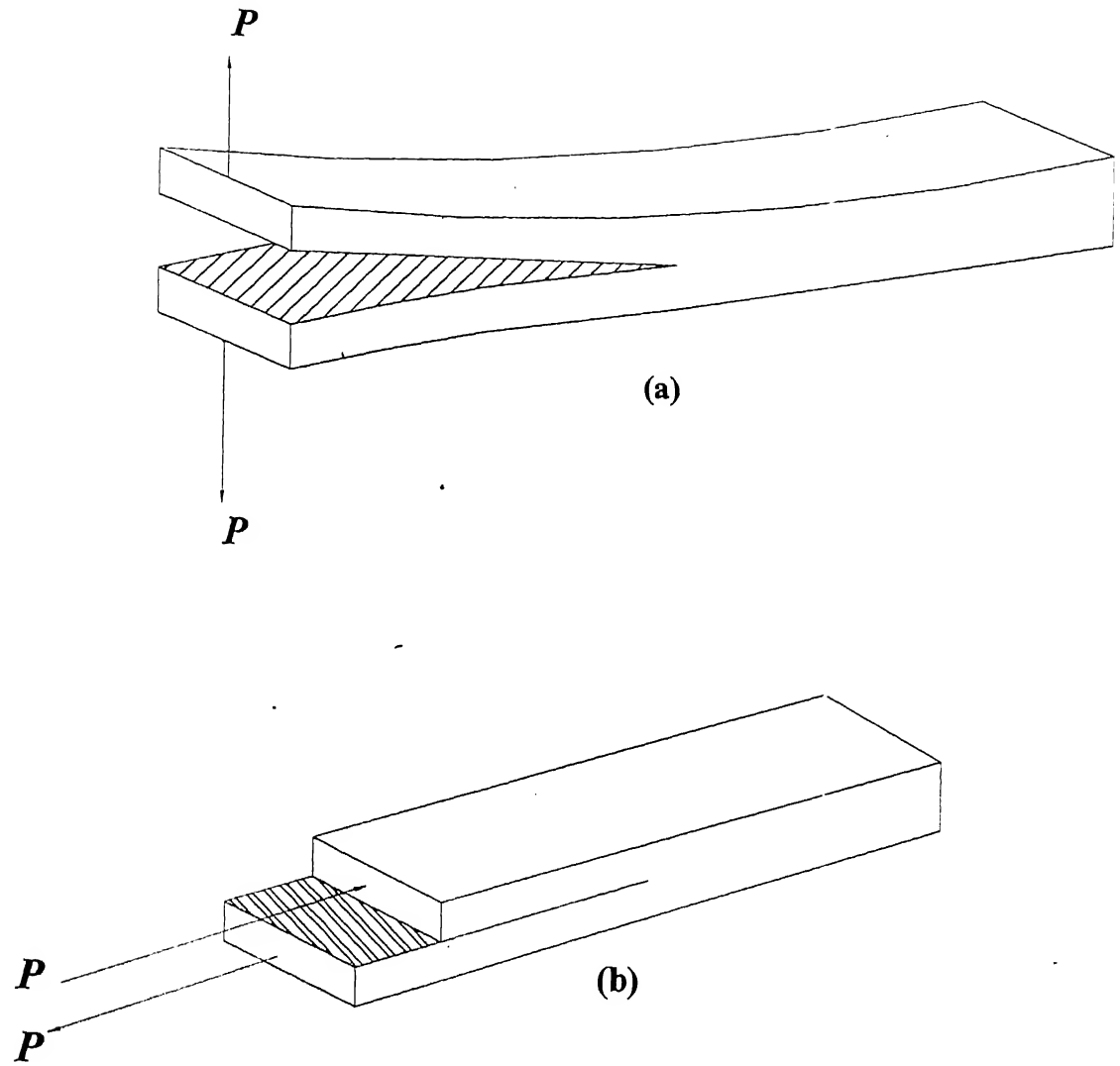


Fig 5.1: (a) Mode I and (b) Mode II

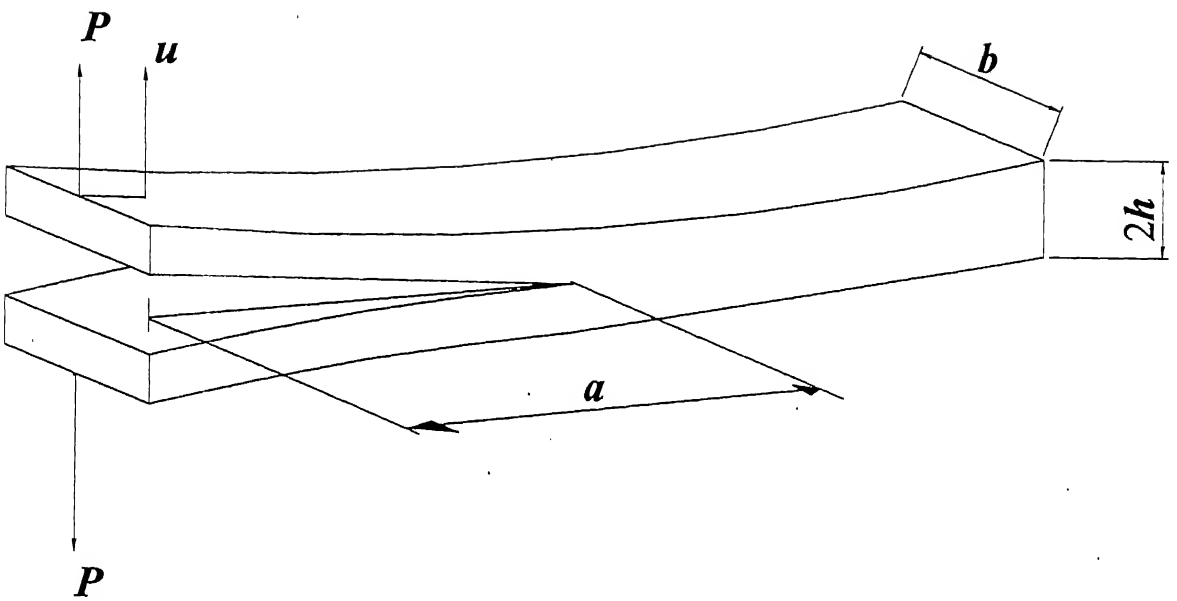


Fig 5.2: DCB specimen

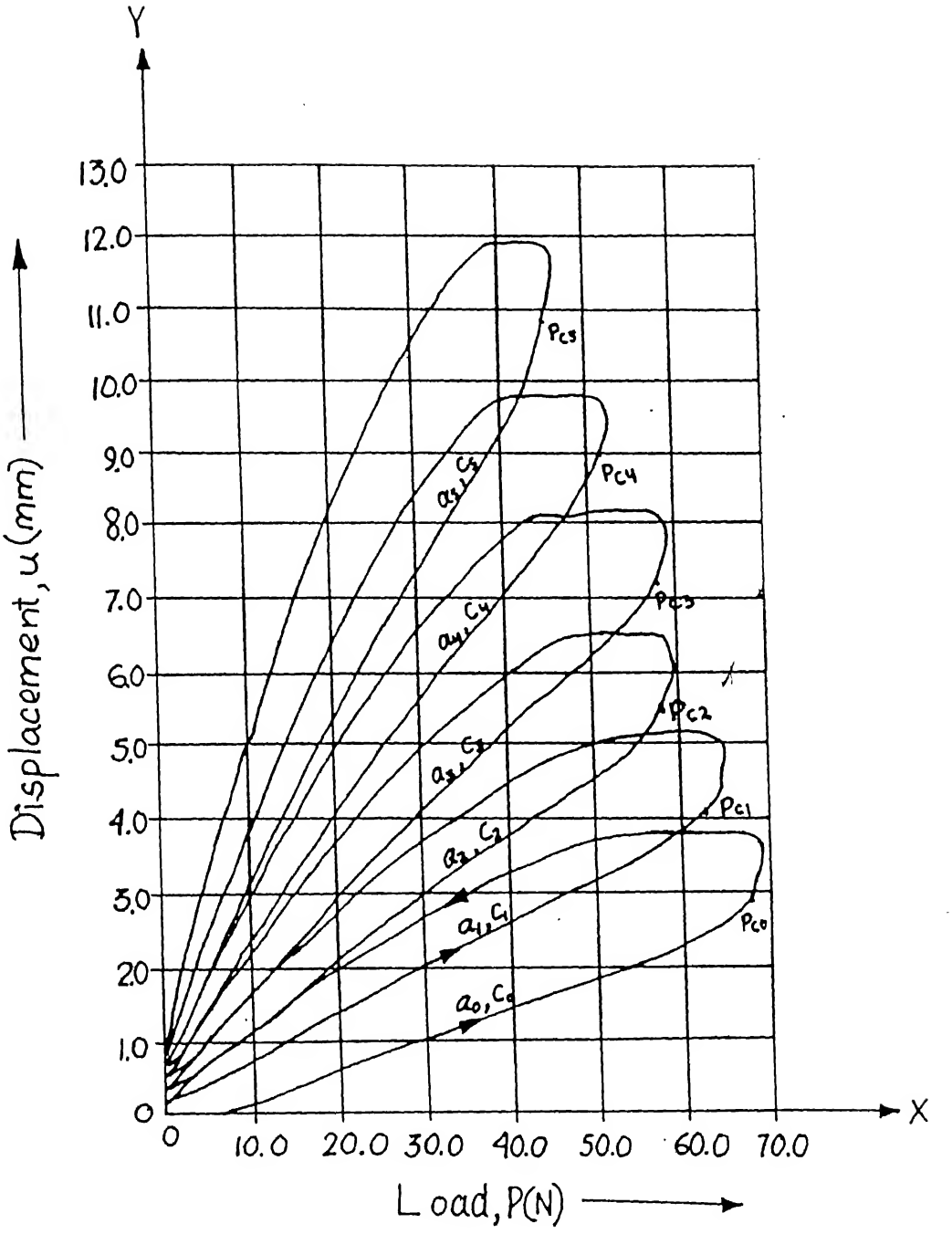
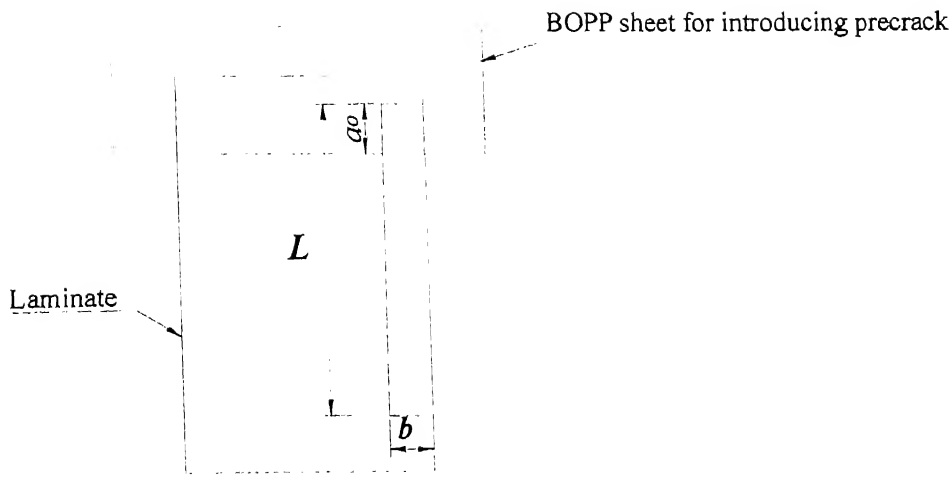
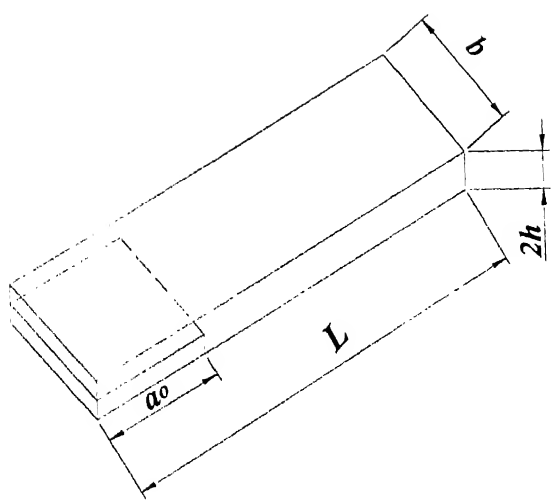


Fig 5.3: Load vs displacement curve



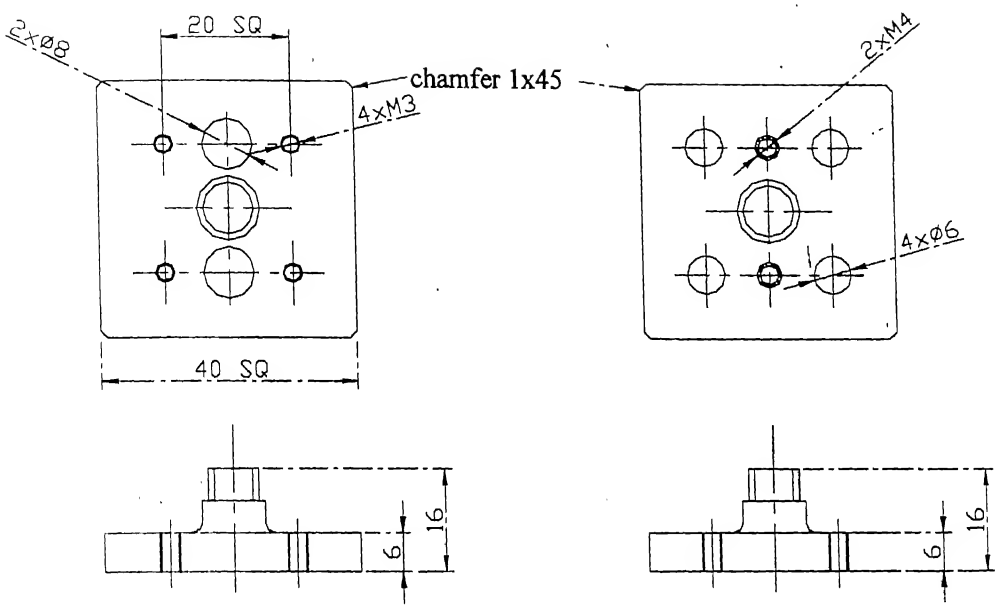
(a)



(b)

Fig 5.4: (a) Preparation of laminates with BOPP sheet at the mid plane

(b) Dimension of specimen prepared from the laminate



Top rectangular tab

Bottom rectangular tab

Fig 5.5: Rectangular tabs used for loading the specimen

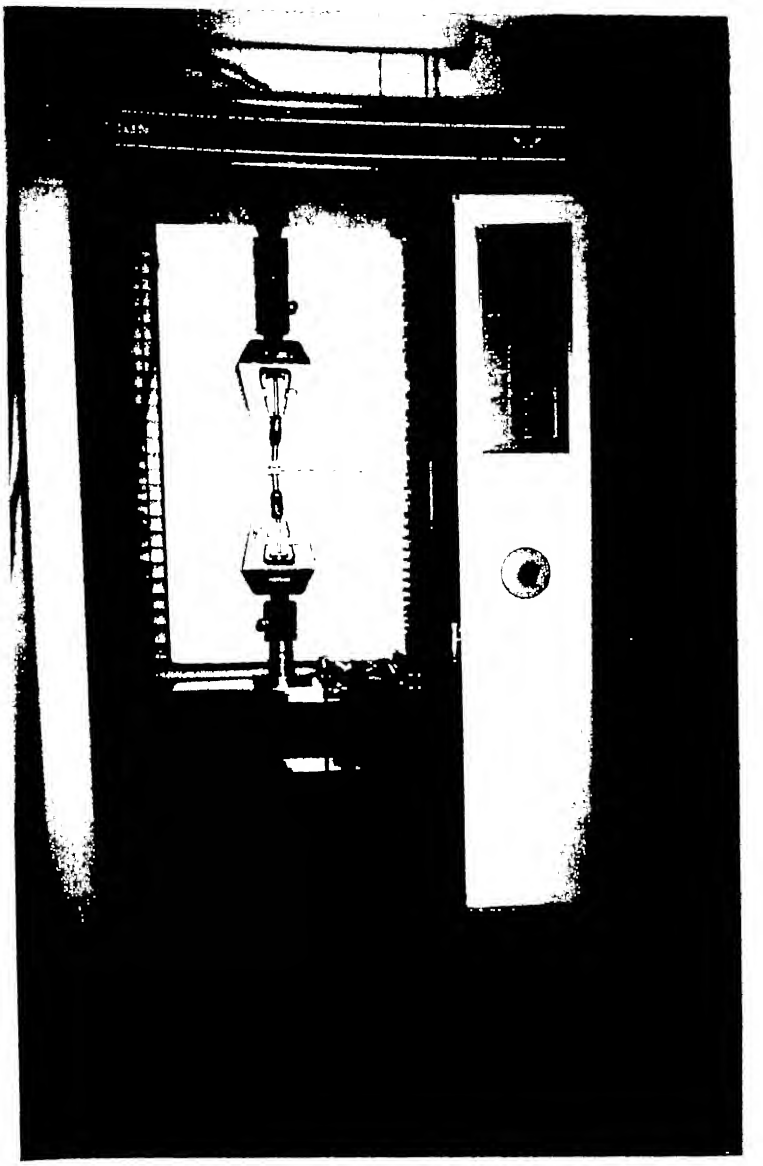


Fig 5.6: Test set-up for mode I test

CHAPTER 6
CHARACTERIZATION OF MATERIALS THROUGH INTERLAMINAR SHEAR TEST

6.1 INTRODUCTION

Interlaminar Shear Strength (ILSS) is an important material property for design of laminated composite structures subjected to transverse loads. The delamination in FRP products can be caused because of shear stress as laminated composites are made of several plies and bonded by polymeric materials.

In the present work, an experimental method known as short beam test method using three point flexural loading is carried out to determine the interlaminar shear strength of a parallel fiber reinforced material. But it only gives the information regarding shear strength but not shear stiffness and shear strain. The objective of present investigation is to determine the interlaminar shear strength for composite beam specimen with 0° fiber orientation along loading direction and compare the interlaminar shear strength of specimen made by the rubber molding method with different rubbers and the conventional method.

6.2 EXPERIMENTAL TECHNIQUE

This section describes specimen preparation, specimen geometry and test procedure to measure interlaminar shear strength.

6.2.1 Experimental method

Short beam method: This method is widely used to determine interlaminar shear strength of fiber reinforced laminated composites. In this method, a three-point flexure specimen

with an appropriate span to depth ratio $2L/d$, is chosen to produce inter-laminar shear failure and shown in Fig 6.1.

Whitney, et al. [1971] have concluded through a theoretical stress analysis that the minimum length, $2L$ to depth, d ratio of the specimen should be 10 and the major Poisson ratio with respect to the specimen edge should be less than unity. If the conditions are not met, a severe stress concentration is developed, which lowers the value of shear strength.

The test setup is made as per specification given in the Dronier Hand book [1987]. It consists of two parts: a base plate and a center load wedge as shown in Figs 6.2 and 6.3 respectively. The wedge of the center load is semicircular at the bottom with the radius of 3 mm. For supporting the specimen, the base plate made of mild steel is used. Its thickness is 15 mm and it has a through slot of 4 mm length and of 16 mm width. A radius of 3 mm is provided on the edges of the slot. Thus, the support points are 10 mm apart when specimen is placed over the slot. Also a recess of 2° is given at the end of the support edges so that the specimen supports only the base plate at the edges. The specimen is made of 2 mm thick to get $2L/d$ of 10. Since homogeneous specimen is used in the present work, simple beam theory can be used for this analysis. The shear stress has a parabolic distribution with the maximum value at the mid-plane and zero on the outer surface of the beam. The maximum shear stress is given by (Agarwal and Broutman, 1990).

$$\tau = (3F)/(4bd) \tag{6.1}$$

where F, b and d are the applied load, width and the depth of the specimen section respectively.

6.2.2 Specimen Material, Geometry and Preparation

The specimen prepared for the present work is by rubber molding methods (using natural, butyl, silicon and polybutadiene rubber sheet) and the conventional method. The specimens are made of 6 layers of glass fiber fabric/epoxy resin in order to get 2 mm thick sheet. The glass fabric fiber, resin and the fabrication process are described in Chapter 4. After the lamina is prepared, it is cut with the help of diamond cutter to the dimension of 20 mm long, 10 mm wide and 2 mm thick as shown in Fig 6.4. Five specimens are cut from single laminate. The test has been conducted on the specimens with 0° orientation of warp fibers along loading direction.

6.2.3 Experimental

The tests are carried out on MTS-810 machine. The test setup is shown in Fig 6.5. The specimen is placed on the base plate and aligned. The tests are conducted in the displacement control mode with a loading rate of 2 mm/min. The load is applied with the centre load wedge. The load vs. displacement graph is shown in Fig 6.6. From the graph one obtains the failure load, which is used in Eq. 6.1 to get the interlaminar shear strength.

6.3 RESULTS AND DISCUSSION

The test is carried out in the specimen with 0° fiber orientation (warp direction) made by (i) conventional method and (ii) rubber molding technique using four different types of rubber namely natural, butyl, silicon and polybutadiene rubber sheets. A typical load and displacement plot is shown in Fig 6.6. The interlaminar shear strength of the specimens prepared by different methods are given in Tables 6.1 to 6.5. From these tables it is clear that the interlaminar shear strength of the specimen made by the conventional method (fiber volume 54%) is 57.2 ± 8.0 MPa and in the rubber molding technique using natural rubber sheet (fiber volume 50%), butyl rubber sheet (fiber volume 51%), silicon rubber sheet (fiber volume 52%) and polybutadiene rubber sheet (fiber volume 52%) are

24.8±5.0 MPa, 59.1±2.5 MPa, 54.9±4.8 MPa and 44.1±5.6 MPa respectively. It is clear from above values that specimens prepared by rubber molding technique using natural rubber have substantially lower interlaminar shear strength by 57% than specimens prepared by the conventional method. The specimen prepared using polybutadiene rubber found to have significantly lower interlaminar shear strength by 24% than specimen prepared by conventional method. However laminates prepared in the rubber molding technique with butyl and silicon rubber found to have marginally higher value compared with laminates prepared by the conventional method and the variations are with in the experimental error.

6.4 CLOSURE

Short beam test is used to determine the interlaminar shear strength. It is concluded that specimens prepared by rubber molding technique using natural rubber have substantially lower interlaminar shear strength by 57% than specimens prepared by the conventional method. The specimen prepared using polybutadiene rubber found to have significantly lower interlaminar shear strength by 24% than specimen prepared by conventional method. However laminates prepared in the rubber molding technique with butyl and silicon rubber found to have marginally higher value compared with laminates prepared by the conventional method and the variations are with in the experimental error.

Table 6.1: Interlaminar shear strength of composite materials made by conventional method ($V_f = 0.54$)

Sl.No	Specimen thickness, *b* (mm)	Specimen width, *d* (mm)	Interlaminar Shear strength, τ (MPa)	Average Shear Strength,τ (MPa)
1	1.78	10.30	62.2	57.2±8.0
2	1.70	10.18	66.7	
3	1.74	9.92	63.4	
4	1.70	9.50	52.1	
5	1.70	9.60	52.3	
6	1.70	9.50	46.5	
Standard deviation σ = 8.0MPa				

Table 6.2 Interlaminar shear strength of composite materials made by rubber molding method (Natural rubber) ($V_f = 0.50$)

Sl.No	Specimen thickness ,*b* (mm)	Specimen width, *d* (mm)	Interlaminar Shear strength, τ (MPa)	Average Shear strength, τ (MPa)
1	1.91	9.90	23.0	24.8±5.0
2	2.12	10.00	33.6	
3	1.98	9.80	20.5	
4	2.04	10.00	27.7	
5	2.10	10.20	22.6	
6	2.06	9.80	21.4	
Standard deviation σ = 5.0 MPa				

Table 6.3: Interlaminar shear strength of composite materials made by rubber molding method (Butyl rubber) ($V_f = 0.52$)

Sl.No	Specimen thickness, *b* (mm)	Specimen width, *d* (mm)	Interlaminar Shear strength, τ (MPa)	Average Shear strength, τ (MPa)
1	1.90	9.9 0	62.9	59.1±2.5
2	1.92	10.20	57.4	
3	1.90	10.10	59.4	
4	1.90	10.10	57.8	
5	1.90	10.20	56.1	
6	1.90	9.90	60.6	
Standard deviation σ =2.5 MPa				

Table 6.4: Interlaminar shear strength of composite materials made by rubber molding method (Silicon rubber) ($V_f = 0.51$)

Sl.No	Specimen thickness, *b*(mm)	Specimen width, *d* (mm)	Interlaminar Shear strength, τ (MPa)	Average Shear strength, τ (MPa)
1	2.00	10.38	53.9	54.9±4.8
2	1.90	10.06	46.0	
3	2.04	10.30	55.7	
4	2.12	10.50	55.1	
5	2.04	10.30	58.9	
6	2.02	10.50	58.9	
Standard deviation σ = 4.8 MPa				

Table 6.5: Interlaminar shear strength of composite materials by rubber molding method (Polybutadiene rubber) (V_f = 0.52)

Sl.No	Specimen thickness, *b* (mm)	Specimen width, *d* (mm)	Interlaminar Shear strength, τ (MPa)	Average Shear strength,τ (MPa)
1	2.00	10.04	53.3	44.1±5.6
2	2.00	10.46	43.8	
3	1.90	10.00	38.7	
4	1.90	9.84	40.5	
5	2.00	10.42	43.9	
Standard deviation σ = 5.6 MPa				

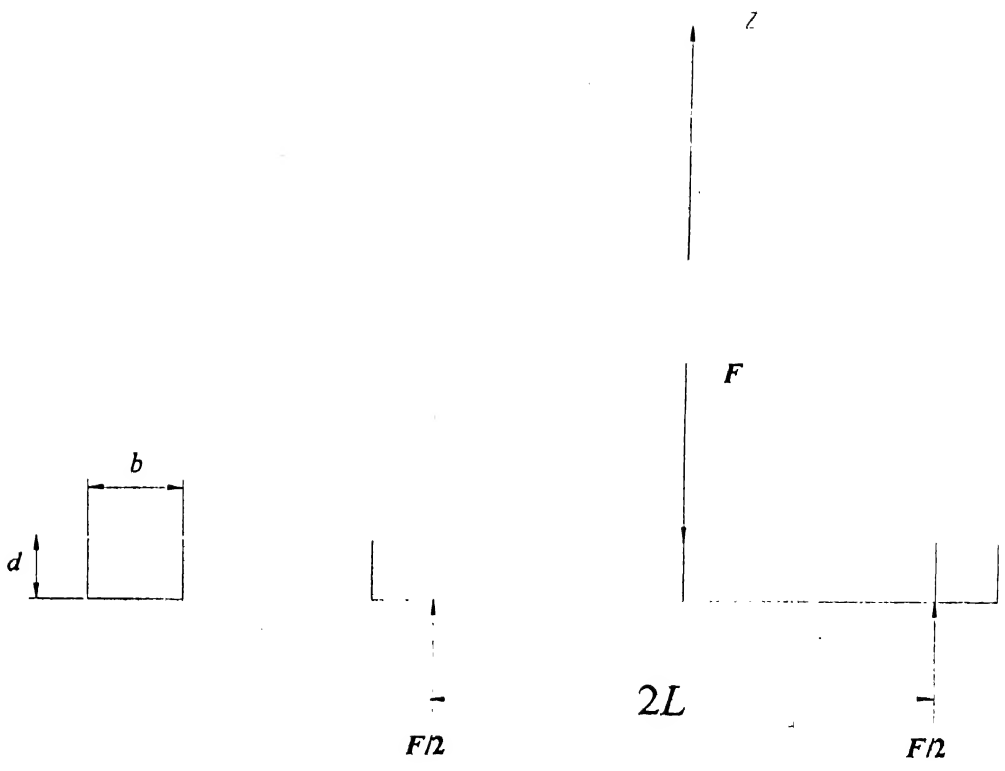


Fig 6.1: Three-Point flexure test (schematic)

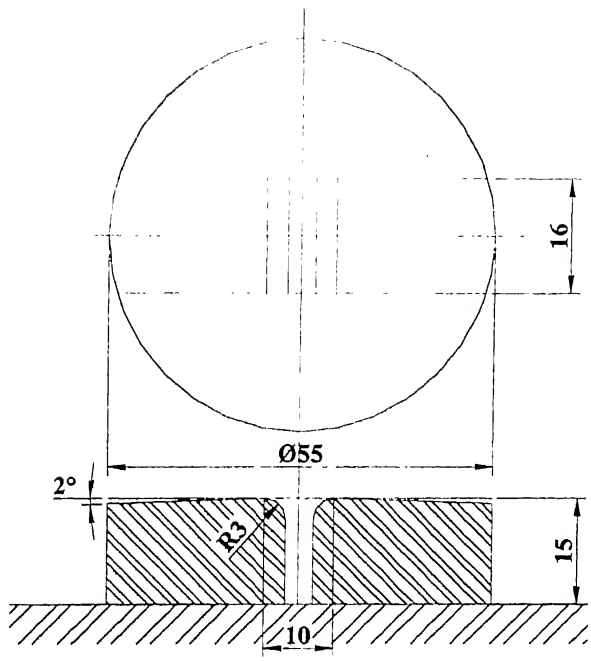
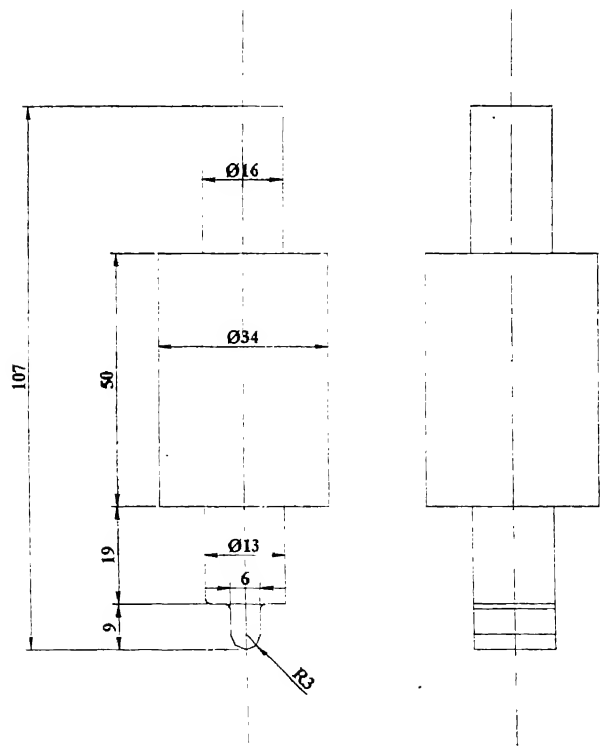


Fig 6.2 Base plate for three-point flexure test



6.3: Central load wedge for three-point flexure test

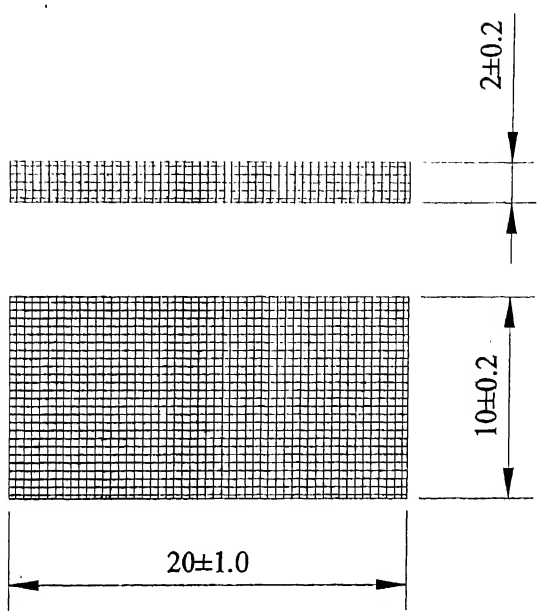


Fig 6.4: Dimension of the specimen

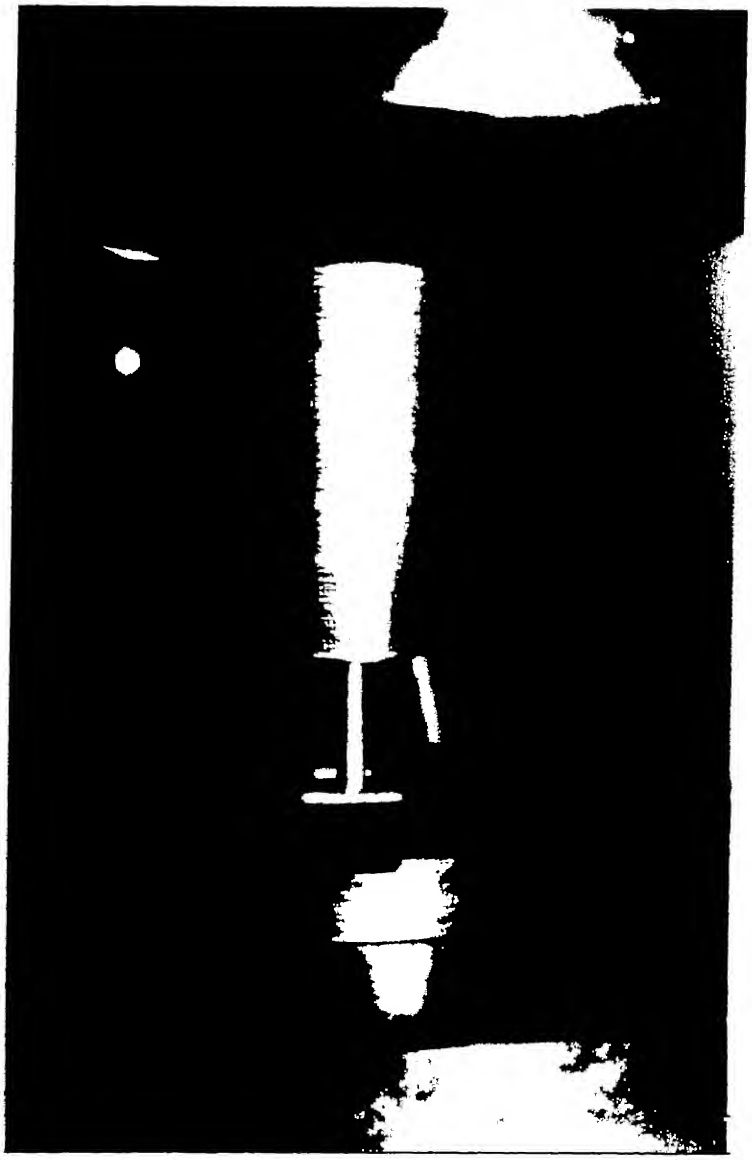


Fig 6.5: Test set up for interlaminar shear test.

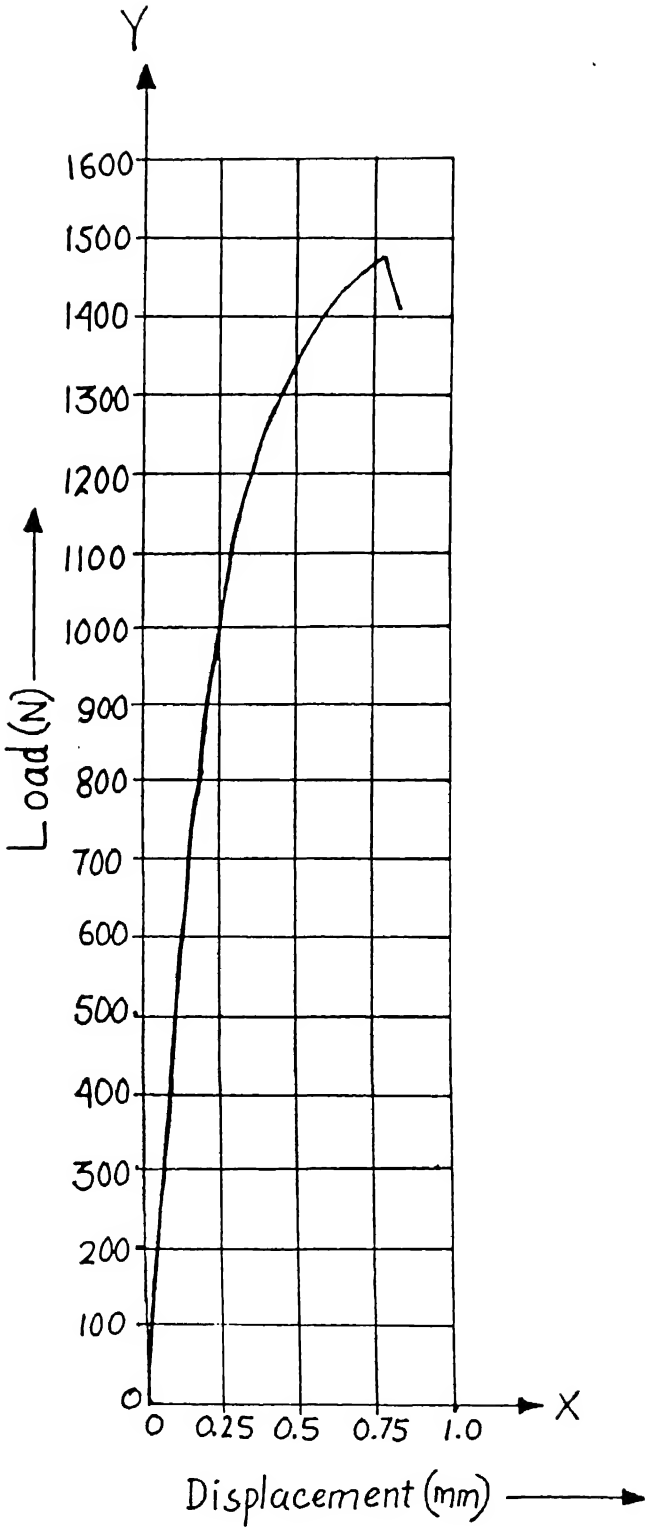


Fig 6.6: Load vs. Displacement curve

CHAPTER 7

CHARACTERIZATION OF MATERIAL THROUGH TENSION TEST

7.1 INTRODUCTION

Tension test is conducted on FRP laminates to evaluate mechanical properties like tensile strength, elastic constants, and percentages of elongation, etc. These properties are very useful for design and analysis of structure made of composite materials. In this chapter, uniaxial test is conducted to determine elastic modulus, tensile strength and the percentage of elongation of the specimen made by the conventional method and rubber molding techniques. The rubbers used in rubber molding technique to prepare the specimen are (i) natural (ii) butyl (iii) silicon and (iv) polybutadiene rubber.

7.2 EXPERIMENTAL

The test method, specimen preparation and test procedure are described here to estimate materials properties.

7.2.1 Test method

The simplest and most widely used mechanical test is static uniaxial test to determine the materials properties like elastic modulus, tensile strength and percentage of elongation of composite laminates and conducted in MTS-810 machine. This is also known as fiber pull test. The machine uses a hydraulic power pack, which provides hydraulic fluid under pressure of 21 MPa for actuator operation. Machine is equipped with hydraulic wedge grips to clamp the specimen and a micro console for its electronic control. The straight sided specimens with 0° fiber orientation (Fig 7.1) along loading direction are used for this test. The straight sided specimen with end tabs (Fig7.2) along with the serrated jaws

of the clamps of tensile machine provides a proper load transmission. The capacity of the machine is 100 kN. A mechanical extensometer with 50 mm gauge length is used to measure strain.

7.2.2 Specimen: Material, Geometry and Preparation

The materials for the specimen preparation are fabric of glass fiber and epoxy resin. The specifications of the glass fiber fabric and the epoxy resin are already described in Ch 4. The geometry of the specimen for tensile test is given in Fig 7.1. The width, length and the thickness of the specimen are 25, 175 and 2.5 mm respectively. The length, width and thickness of end tabs are 30, 25 and 2 mm respectively (Fig 7.2). To prepare specimen with 0° orientation along loading direction and 2.7 mm thickness 8 layers of bidirectional glass fiber are used along with epoxy resin.

The laminates are prepared by rubber molding technique (using natural, butyl, silicon and poly butadiene rubber sheets) and conventional method. The process of fabrication of laminates is described in the Ch 4. After the preparation of the laminate in conventional and rubber molding techniques, the laminates are cut to the specified dimension using diamond impregnated wheel cutter.

The end tabs used in the specimen with thickness of 2 mm are prepared from 6 layers of bidirectional glass fiber fabric and epoxy resin. The end tabs are cut from these laminates to the required geometry (Fig 7.2). Then the end tabs are bonded to the specimen using epoxy resin. After bonding the end tabs, specimens are marked at the center of the specimen to place the extensometer.

5.2.3 Experimental

The specimen is clamped in the hydraulic wedge grip of the MTS-810 machine up to the depth of 30 mm from both ends. The proper alignment of the specimen in the jaw is

assured as minor change in the direction of pull gives an incorrect value. The extensometer with 50 mm gauge length is placed at the center of the specimen. The test setup for the tensile test is shown in Fig 7.3. The tests are conducted in displacement control mode with loading rate of 1 mm/min. The applied load is measured with the help of load cell and strain is measured with the help of extensometer. A typical load vs strain graph (shown in Fig 7.4) is obtained from the plotter of the MTS machine. Tensile strength, modulus of elasticity in longitudinal direction and the percentage of elongation are calculated from this graph. The slope of the graph is linear initially but as load on the specimen increases the fiber starts breaking and the curve becomes non-linear. The slope of the linear curve is used to calculate the modulus of elasticity. The tests are conducted on 5 specimens prepared by each technique. The photograph of broken specimen is shown in Fig 7.5.

7.3 RESULTS AND DISCUSSION

The tension test has been conducted on the specimen prepared by conventional and rubber molding methods (using natural, butyl, silicon and poly butadiene rubber sheets). The volume fraction of fiber is 52 to 54% using pressure of 0.5 MPa. The results of mechanical properties using both techniques are given in the Tables 7.1 to 7.5. The specimens are found to fail at the center portion of the specimen and the facture line makes 45° to the line of loading. A typical load vs. strain graph is shown in Fig 7.4. It is linear at the low load but non-linear at higher load due to breaking of fiber.

The average value of tensile strength of the specimen made by the conventional method is 312 ± 35 MPa. But the average value of tensile strength of the specimens prepared by rubber molding technique using natural, butyl, silicon and poly butadiene rubber sheets are 309 ± 28 MPa, 346 ± 17 MPa, 342 ± 32 MPa 338 ± 6 MPa respectively.

But the average value of tensile elastic modulus of the specimen made by the conventional method is 19.0 ± 1.0 GPa, where as the average value of elastic modulus

of the specimen prepared by rubber molding technique and using natural, butyl, silicon and poly butadiene rubber sheets are 19.8 ± 0.2 GPa, 20.9 ± 0.6 GPa, 20.9± 0.7 GPa and 19.5 ± 0.82 GPa respectively.

Similarly the average value of percentage of elongation of the specimen made by the conventional method is 2.0 ± 0.1 %. The average value of percentage of elongation of the specimen prepared by rubber molding technique, using natural, butyl, silicon and poly butadiene rubber sheets are 2.0 ± 0.2 %, 2.2 ± 0.1 %, 2.1 ± 0.1 % and 2.1 ± 0.1 % respectively.

It is clear from the above observation that the tensile strength of specimens made by rubber molding method are marginally higher than the conventional method except the value of the tensile strength of the specimen prepared by natural rubber sheet, which has equal value than specimen prepared by conventional method. The elastic modulus of the specimen prepared by rubber molding technique using butyl and silicon rubber sheets marginally higher by 9 % each than the specimen made by conventional method. These variations can be due to small variation of volume fraction of fiber between specimens prepared in different methods. However, the specimen prepared by using natural and polybutadiene rubber sheets gives equal value of elastic modulus that of specimen prepared by conventional method. The variations of percentages of elongation of the specimens made by rubber molding technique and conventional methods are negligible and average values are with in experimental error bands.

7.4 CLOSURE

The fiber pull test or tension test is performed on the specimen made by conventional and rubber molding techniques (using natural, butyl, silicon and polybutadiene rubber sheet). The tensile strength of specimens made by rubber molding method are marginally higher than the conventional method except the value of the tensile strength of the specimen prepared by natural rubber sheet, which has equal value that of specimen prepared by

conventional method. The elastic modulus of the specimen prepared by rubber molding technique using butyl and silicon rubber sheets marginally higher by 9 % each respectively than the specimen made by conventional method. The variations of percentages of elongation of the specimens made by rubber molding technique and the conventional methods are negligible and average values are with in experimental error bands. The percentages of elongation of the specimen prepared both the techniques are within error bands.

Table 7.1: Mechanical properties of FRP laminate made by conventional method (V_f=0.52)

Specimen No.	Width (mm)	Thickness (mm)	Tensile strength (MPa)	Modulus of elasticity (GPa)	% of elongation
1	24.86	2.72	284	18.2	2.1
2	24.96	2.68	311	20.1	2.0
3	24.86	2.70	366	18.9	2.0
4	24.92	2.72	277	20.0	1.8
5	24.92	2.68	321	18.1	2.1
Average tensile strength = 312 ± 35MPa Average modulus of elasticity = 19.1 ± 1.0 GPa Average elongation =2.0 ± 0.1 %					

Table 7.2: Mechanical properties of FRP laminates made by rubber molding technique (using natural rubber sheet) (V_f=0.54)

Specimen No.	Width (mm)	Thickness (mm)	Tensile strength (MPa)	Modulus of elasticity (GPa)	% of elongation
1	24.90	2.68	308	20.6	2.1
2	25.04	2.66	314	19.9	2.0
3	24.86	2.68	335	20.0	2.0
4	25.12	2.68	263	19.9	1.9
5	25.02	2.66	324	18.5	1.9

Average tensile strength =309 ± 28 MPa

Average modulus of elasticity =19.8 ± 0.2 GPa

Average elongation =2.0 ±0.1 %

Table 7.3: Mechanical properties of FRP laminate made by rubber molding technique (using butyl rubber sheet) (V_f=0.54)

Specimen No.	Width (mm)	Thickness (mm)	Tensile strength (MPa)	Modulus of elasticity (GPa)	% of elongation
1	24.96	2.68	319	20.6	2.3
2	25.06	2.66	350	20.2	2.1
3	25.18	2.66	343	21.8	2.3
4	25.18	2.68	361	20.9	2.0
5	24.96	2..66	358	21.2	2.1

Average tensile strength = 346 ±17 MPa

Average modulus of elasticity =20.9 ± 0.6 GPa

Average elongation =2.2± 0.1 %

Table 7.4: Mechanical properties of FRP laminate made by rubber molding technique (using silicon rubber sheet) (V_f =0.54)

Specimen No.	Width (mm)	Thickness (mm)	Tensile strength (MPa)	Modulus of elasticity (GPa)	% of elongation
1	24.76	2.68	353	20.2	2.1
2	24.76	2.66	286	21.9	2.3
3	24.72	2.66	354	21.4	2.2
4	25.12	2.68	360	21.2	2.0
5	25.04	2.70	358	20.1	2.1

Average tensile strength =342 ± 32 MPa

Average modulus of elasticity =20.9 ± 0.7 GPa

Average elongation =2.1 ± 0.1 %

Table 7.5: Mechanical properties of FRP laminate made by rubber molding technique (using polybutadiene rubber sheet) (V_f =0.53)

Specimen No.	Width (mm)	Thickness (mm)	Tensile strength(MPa)	Modulus of elasticity(GPa)	% of elongation
1	25.12	2.68	341	20.6	2.0
2	24.96	2.72	339	19.5	2.1
3	224.96	2.70	330	18.2	2.2
4	25.24	2.68	333	19.5	1.9
5	24.94	2.68	346	19.9	2.2

Average Tensile Strength = 338 ± 6 MPa

Average Modulus of Elasticity =19.5 ± 0.8GPa

Average Elongation =2.1 ± 0.1 %

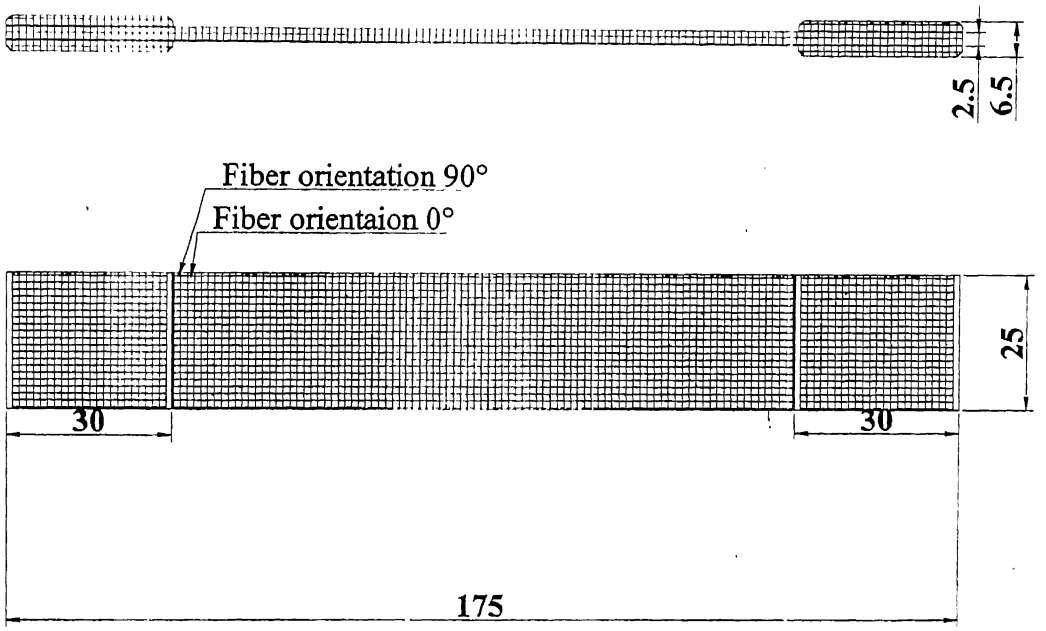


Fig 7.1: Tensile test specimen with end tabs

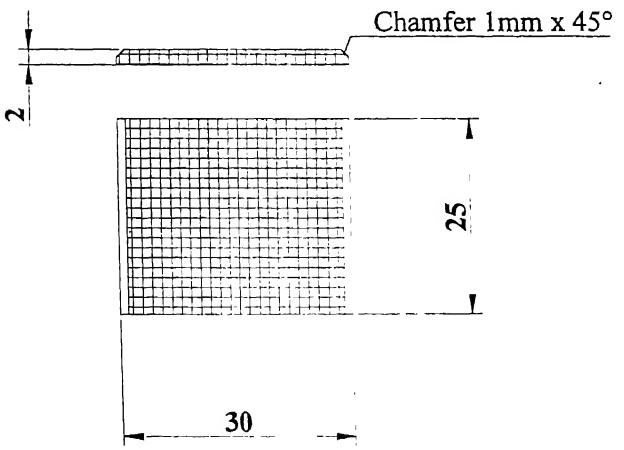


Fig 7.2: End tabs (GPRP) for tensile test specimen

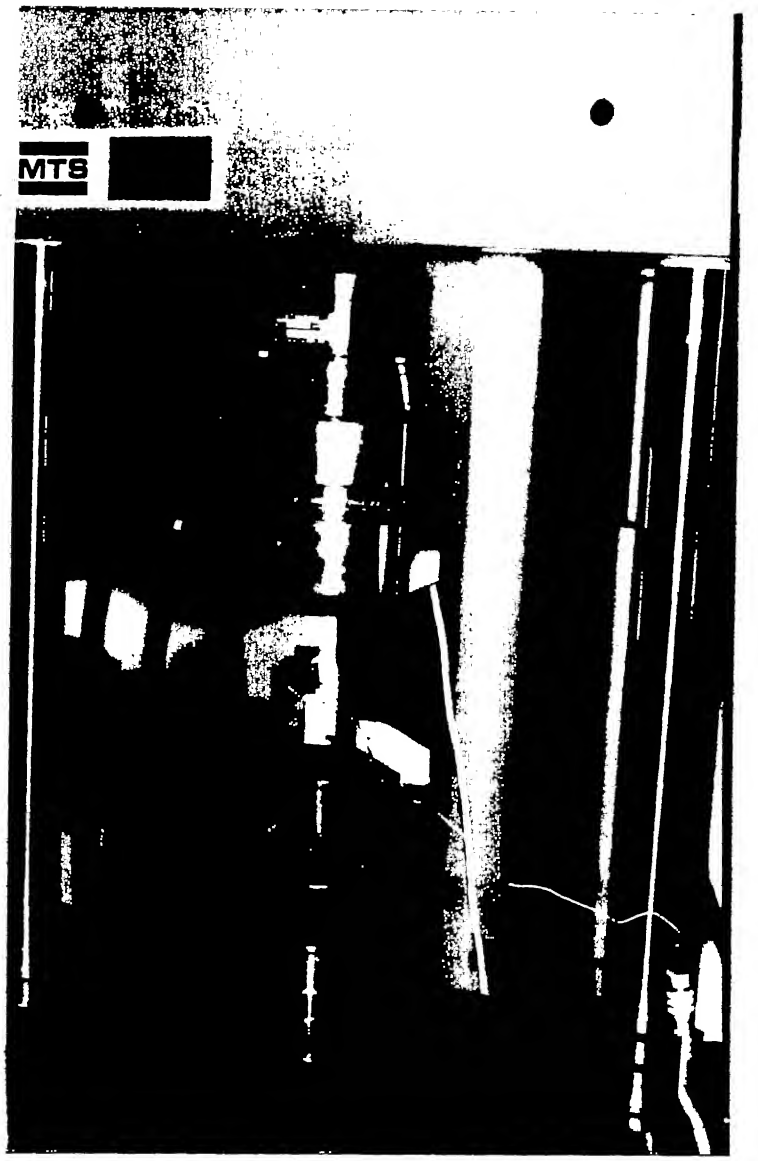


Fig 7.3: Test set up for the tensile test

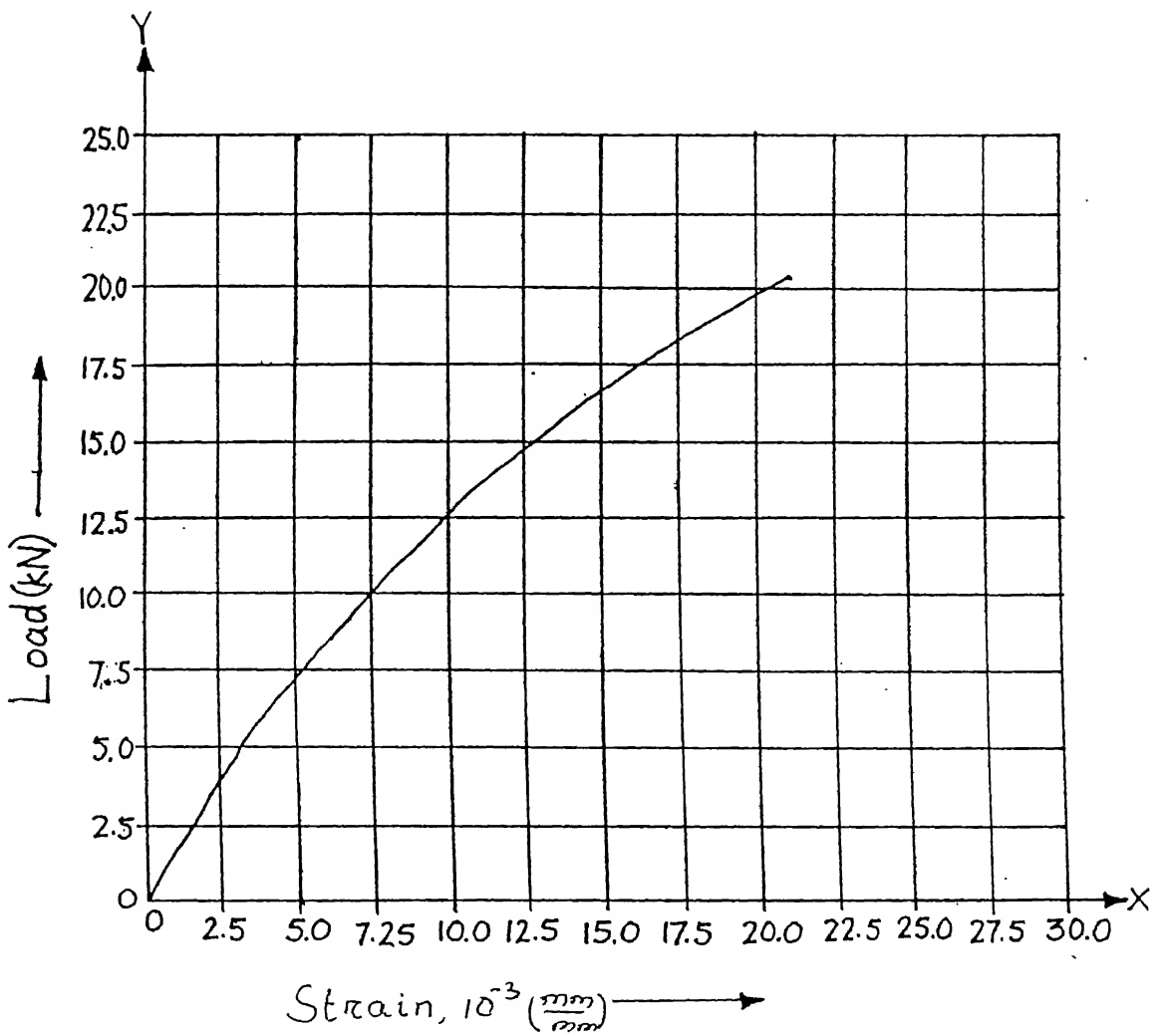


Fig 7.4: A typical load vs. strain curve

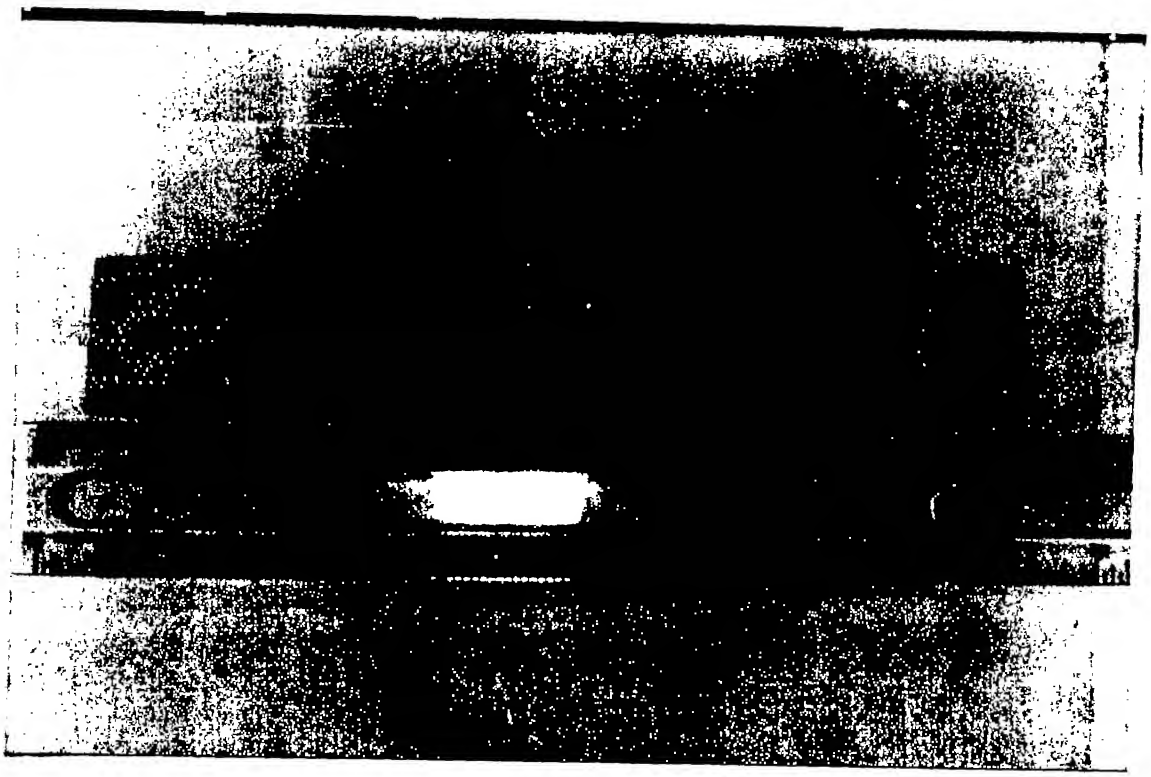


Fig. 7.5: Broken tensile test specimen

CHAPTER 8
CONCLUSIONS

Rubber molding technique is a method to fabricate FRP products. This technique is similar to matching die set method. But in rubber molding technique, the die is of hard metal like steel and punch is of flexible material like rubber.

In rubber molding technique, rubber and polymer (matrix) are in direct contact during curing of a FRP component. There is possibility of certain chemical reaction between both polymers at operating temperature and pressure. To find out the feasibility of using rubber punch in a given resin system, four varieties of rubbers are tested with epoxy and polyester resin. The rubbers used in the test are (i) natural, (ii) butyl, (iii) silicon and (iv) polybutadiene. It is found epoxy resin cures well, but polyester resin does not cure with any of the above rubbers. Therefore epoxy resin and glass fiber reinforcement is used to prepare a product "pump cap" using above four varieties of rubber punch.

To check feasibility of different rubber punches in rubber molding technique, the product is compared qualitatively with four varieties of rubber used in present work. The materials are characterized through burn test for finding volume fraction and void content of different portion of the product, coin test to check product delamination and electron microscopy for microstructure. The performances of all rubbers are same with respect to above mention properties.

Various mechanical tests are conducted to characterize FRP specimens prepared by conventional matching die set and rubber molding techniques. Again in rubber molding technique same four varieties of rubber sheets are used to prepare specimen.

In interlaminar fracture toughness test, specimens prepared using butyl and silicon rubber sheet have interlaminar fracture toughness equivalent to those of specimen prepared by the conventional method. But the specimen prepared by using natural rubber and polybutadiene found to have significantly lower interlaminar fracture toughness compared to the specimen prepared by conventional method.

In interlaminar shear test, specimen prepared using natural rubber has lower interlaminar shear strength than specimens prepared by conventional method. However laminates prepared in rubber molding technique with butyl and silicon rubber found to have marginally higher value compared with laminates prepared by conventional method.

In tension test, specimens made by rubber molding method have marginally better value of tensile strength than specimen prepared by conventional method except specimens prepared using natural rubber. The elastic modulus of the specimen prepared by rubber molding technique using butyl and silicon rubber sheets found to have slightly higher value than the specimen made by conventional method.

Thus FRP products prepared using rubber molding technique gives comparable mechanical properties with conventional method except the product made using natural rubber. Polybutadiene rubber is cheaper than butyl and silicon rubbers. Therefore polybutadiene rubber in rubber molding technique can be used to fabricate fiber reinforced plastic products for aerospace, automobile as well as for daily life products.

REFERENCES

1. Agrawal, B.D. and Broutman, L.J., "Analysis and performance of fiber composite" John Wiley and sons, Inc., New York, (1990).
2. Bacom, W.D., Britner, J.L., Moultar, R.J. And Siebert, A.R., "The Interlaminar Fracture of Organic-matrix Woven Reinforcement Composites", *Composite* (1980).
3. Choi, N. S., Kinloch, A. J. and Williams, J. G. Delamination Fracture of Multidirectional Carbon-Fibre/Epoxy Composites under Mode I, Mode II and Mixed-Mode I/II Loading, *Journal of Composite Materials*,Vol. 33(1999), p73
4. Devitt, D.F., Schapery, R.A. and Bradley, W.L., "Method of Elastic and Viscoelastic Composite Materials".,*J.Composite Materials*,Vol. 14(1980), p270.
5. Dronier Handbook, DOL 63, part1, p1.
6. Dronier Handbook, DOL 65, part4, p1.
7. Dronier Handbook, DON 128, part1, p23.
8. Edwards, K. L. "An overview of the technology of fiber-reinforced plastics for design purposes", *Materials & Design*, Vol.19 (1998), p1
9. Han, K. S. and Koutsky, J., " The interlaminar Fracture Energy of Glass fiber Reinforced Polyester Composites" , *J. Composite Materials*, Vol.15(1981), p371.
10. Hill, R. G., "Evaluation of Elastic Modulii of Bilaminate Filament Wound Composites", *Experimental Mechanics*, Vol.8(1968), p75.
11. Hollaway, L., "Handbook of Polymer Composites for Engineering" Woodhead Publishing Ltd.,Cambridge England(1994).
12. Hsu, P.W. and Herakovich, C.T., "A perturbation Solution for Interlaminar stresses in Composite laminates", ASTM 4th Conference on Composite Materials:Testing and Design, Vallei Forge,PA,May (1976).
13. Hsu, P.W. and Herakovich, C.T., "Edge Effects in Angle-Ply Composite Laminates", *J.Composite Materials*, Vol.11 (1977), p422
14. Keary, P. E., Sharr, C., Ilcewicz, L. B. and Trostle, J. Mode I Inter laminar Fracture Toughness of Composites using Slender Double Cantilevered Beam Specimens, *Journal of Composite Materials*, Vol.19, (1985),p154

15. Kumar, P. *Elements of Fracture Mechanics*, Wheeler publishing, India (1999).
16. Kumar P., "Polymer Composite Products from continuous fibers" Indo-Japan Seminar on advance Manufacturing, Gangtok Sikkim, (Feb.21-23, 2000).
17. Kumar, P., Goel, B. and Bandhopadhyay, J., "Unidirectional Prepreg machine –An indigenous Development" *Journal of Institution of Engineers*, India, Vol.75, (1995)
18. Kumar, S. "Development of Techniques to Manufacture FRP Products using Rapid Tooling", *M.Tech. Thesis*, IIT Kanpur. (2001).
19. Lantz, R.B., "Boron-Epoxy Laminate Test Methods", *J. Composite Materials*, Vol. 3 (1969), p 642
20. Mall, S., Law, G.E. and Katouzian, M., "Loading rate effect on Interlaminar fracture toughness of a Thermoplastic Composite", *J. Composite Materials*, Vol.21 (1987), p 569.
21. Mangalgiri, P. D., "Composite Materials for Aerospace application", *Bull. Material Science* ,Indian Academy of Sciences, Vol. 22,(1999)
22. Morom, G., Roman, I., Harel, H., and Rosesaft, M., "The Characterization of Mode I and Mode II Delamination Failures in Fabric Reinforced Laminates", VIICCM, Vol. 3, (1987), p 3265.
23. Mallon, P.J. and Obradaigh, C. M., " Development of pilot autoclave for polymeric diaphragm forming of contnous fiber reinforced thermoplastics", *J. of Composites*, Vol 19, (1988), p 37.
24. O'Brien, T. K. "Characterization of Delamination Onset and Growth in a Composite Laminate", ASTM STP, Vol. 775 (1982), p.140-167
25. Pagano, J.C. and Halpin, "Influence of End Constraints in the Testing of Anisotropic Bodies", *J. Composite Materials*, Vol. 2(1968), p18
26. Pagano, N. J. and Pipes, R. B., "Some Observations on the Interlaminar Strength of Composite laminates", *International J. Mechanical Sciences*, Vol. 15 (1973), p 679
27. Parkyn Brian, "Glass Reinforced Plastics" Iliffe Books, London, (1970)
28. Pipes, R. B. and Pagano, N. J., "Interlaminar Stresses in Composite Laminates under uniform axial extension", *J. Composite Materials*, Vol. 4 (1970), p 538

29. Rybicki, E. F., "Approximate three dimensional solution for symmetric laminates under inplane loading", *J. Composite Materials*, Vol. 5 (1971), p 354

30. Shah, T. K. "Development of Rubber Pressure Molding to Fabricate FRP Components", *M. Tech. Thesis*, IIT, Kanpur, India (2001).

31. Stringer, L.G., " Optimization of the wet lay-up/ vacuum bag process for the fabrication of carbon fiber epoxy composites with high fiber fraction and low void contents", J. Composite Materials, Vol 20.(1989),p 441.

32. Vangerko, H., "Composite Tooling for Composite Components", Composites, Vol.19 (1988). p.481

33. Waddoups , M. E., "Characterization and Design of Composite Materials", Composite Materials Workshop,Tsai,S.W., Halpin, J. C. and Pagano, N. J., Eds.,Technomic, Stanford , Conn.,(1968).